### ON FARM ETHANOL DEWATERING:

### DETAILED DESIGN

OF

### DISTILLATION UNIT

By

### ANURADHA MUKHERJEE

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Thesis Approved:

Dr. James R. Whiteley
Thesis Adviser
Dr. Danielle Bellmer
Dr. Haathar Fahlankamn
Dr. Heather Famenkamp
Dr. A. Gordon Emslie
Dean of the Graduate College

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### CHAPTER I

### **INTRODUCTION**

Renewable or alternative energy encompasses many sources of naturally available resources. Solar, wind, and biomass are examples of alternative sources of energy. As seen in Fig 1.1, among the various sources of renewable energy available, biomass accounts for a large portion of the U.S renewable energy supply.



Fig 1.1: U.S Energy consumption supply 2008, Source EIA Office of Coal, Nuclear, Electric and Alternate Fuels.

Apart from being used in industrial electricity generation, biomass has significant contributions to the liquid fuel sector. Unlike other renewable sources which cannot be converted to liquid fuels, ethanol and biodiesel, biobutanol are some of the fuel products that originate directly from biomass. Ethanol is the most widely used replacement in the transportation fuels sector. The production of fuel grade bioethanol in the US in the year 2008 was 9 billion gallons (RFA 2009) and it is growing steadily with each progressing year. These high volumes of biofuel production are governed to a large extent by policies introduced to encourage production. The most recent and perhaps the most important policy, in terms of impact on the production and consumption of fuel ethanol, is the Energy Independence and Security Act (EISA) of 2007. Signed into law on Dec 19<sup>th</sup>, 2007, the EISA established the Renewable Fuels Standard (RFS). The RFS requires an annual production of 36 billion gallons of ethanol by 2022 with 15.2 billion gal/year production by 2012. This is the primary reason which makes the current scenario different from ones seen in the past. The production targets of ethanol have now been legislated and thus there is an assured need for bioethanol as a transportation fuel.

One of the stipulations of the EISA, which could have a significant impact on the biofuel production landscape, is the requirement for a major portion of ethanol to come from advanced cellulose based feedstocks. This portion should account for 21 billion gal/year ethanol production by year 2022. From the above amount 16 billion are expected from cellulosic material and the rest from other sources (EIA 2008).

At present, the use of corn accounts for a significant share of the bio-ethanol landscape and has seen the greatest amount of commercial activity. However, this has caused worldwide concerns. Some of the issues include a low corn energy return with only 24% gain in the energy output (Shapouri et al. 1995), large water and fertilizer requirements (Patzek 2004), and corn's position as a food crop (Abbott et al. 2008). In the wake of these concerns and other environmental, financial and national security reasons, the production of ethanol from corn has a cap of 15 billion gal/year beyond the year 2015.

Some of the newer feedstocks being considered are sugar based crops such as sugarcane and sugar beets. Starch based crops such as maize, wheat or barley, cellulosic

feedstocks such as wood chips, solid wastes and agricultural wastes are some of the other new feedstocks being explored as sources of ethanol. One of the sugar based crops being considered in the US is sweet sorghum or *Sorghum bicolor (L) Monech*. Sweet sorghum offers a variety of benefits as a raw material for production of bio-ethanol. It was identified as a preferred renewable energy crop with "long range appeal" as far back as 1978 (Nathan 1978). The nutrient requirement of sweet sorghum is low. It can be grown in some of the harshest environments and is resistant to droughts (Bellmer et al. 2007). It does not conflict with the cultivation of food crops.

The benefits of using sweet sorghum as feedstock are partially offset by the need to initiate fermentation immediately to minimize the loss of microbial conversion of the sugars to non-ethanol products. The on-farm concept for production of ethanol from sweet sorghum germinates here. An on-farm facility for separating and dewatering ethanol can be conceptualized as a small scale unit, operated by the farmer which uses a decentralized model for ethanol production. Fermentation can take place in "bladders" placed on the sweet sorghum plot. The process is simple as the biomass being treated consists of easily fermentable six-carbon sugars which once fermented, give 6-10 vol% ethanol stable for long-term storage and future processing (Kundiyana 2006).

For economic reasons, dewatering to remove most of the inherent water, at the site of fermentation becomes essential. The main advantage to having a decentralized on-farm ethanol dewatering facility is avoidance of transporting water (90+ % of the fermentation product) to a centralized processing facility. One must keep in mind though that any large scale unit has economy of scale in their favor. It is due to this reason there has been activity worldwide in sweet sorghum bioethanol related developments.

Currently several operations have either been set up or are in the pipeline in various locations around the world which include large plants based on central collection of sweet sorghum feedstock (PRAJ 2009).

A small scale operation has advantages though primarily due to the simplicity of its operation. In addition and more importantly, economic opportunities for agricultural producers are greatly enhanced. It would provide small farmers who have low returns per acre an opportunity to increase the size of their business. Furthermore, a farmer with substantial land or a co-operative of farmers, can perhaps successfully produce ethanol from sweet sorghum by sharing the large costs incurred for processing the ethanol to fuel (Fryer 2008).

Decentralized production of ethanol may offer other advantages as well. First, disbursement of the byproducts can be simplified and some of these, like the distillation bottoms, may be used directly on the farm. Sweet sorghum stillage is known to contain fertilizer value of 0.2% N, 0.22%  $P_2O_5$  and 0.3%  $K_2O$  and may possibly be discharged into the fields without further treatment (Grassi 2005). Transportation costs are also highly reduced, since only the final product is being shipped.

Sorganol<sup>TM</sup>, which is ethanol produced using sweet sorghum as feedstock, is central to the concept of on-farm ethanol dewatering and is described in detail in Fig 1.2. The process involves harvesting and pressing the juice out of the stalks on the farm, fermenting the sugar juice in bladders followed by the ethanol separation step to produce fuel grade ethanol on farm.



Fig 1.2: The Sorganol Process as envisioned by Mr. McLune, 2002 (adapted from www.sorganol.com)

The team at the School of Bio-systems & Agricultural Engineering at Oklahoma State University has demonstrated the ability to harvest, express, and ferment sweet sorghum on-farm. (Bellmer et al. 2006). Their results on small scale in field fermentation results demonstrate that fermentation can be carried out without temperature control, presterilization or adjustments to the pH or nutrients. The fermentation product is an aqueous 6 - 10 wt % ethanol product (Bellmer et al. 2006; Bellmer et al. 2007). Large scale trials of the same have been scheduled for the near future. The high concentration of water (90+ wt %) in the fermentation product provides a strong incentive to perform on-farm dewatering in lieu of costly transport to a central processing facility.

The design of such an on-farm facility poses an interesting technical challenge. The production of fuel grade bioethanol corresponds to 99.5 wt% ethanol content (ASTM 2009), suitable for use in vehicles. This process however is complicated by the presence of an azeotrope at 95 wt% ethanol. The technology to separate ethanol from water, beyond the azeotrope to manufacture 200 proof alcohol, has been established over the years for large scale processing (RFA 2007). These centralized bio-ethanol separation systems are large scale process plants capable of producing 100+ gpm of fuel grade ethanol. There are multiple columns including the beer stripper and the rectifier in arrangements depending on the end use of the ethanol. For anhydrous ethanol production, molecular sieve is the most commonly used technology to dewater past the azeotrope. Molecular sieve dewatering units are capable of handling as much as 800 gal/h 192 proof ethanol feed (K.A. Jacques et al. 2003).

Unfortunately, these designs cannot be applied without significant modification to the on-farm case. The two main factors that drive the technical design of such an operation are its scale and operational environment. Unlike industrial scale facilities, onfarm units operate at low feed rates of about 250 gallons per hour (Nellis et al. 2000). The corresponding ethanol production rate of less than 1 gpm is more than two orders of magnitude less than a commercial facility. It is also unlikely that an on-farm facility will have access to traditional utilities such as steam or cooling water. Therefore, the design of an on farm unit will differ from its industrial counterpart.

The on-farm or small scale production of ethanol presents a set of novel technological challenges and opportunities from a design and engineering stand point, the success of which lies in it being economical and easy to operate by on-farm personnel. To make on-farm ethanol from sweet sorghum a reality, the design of the separation system is the first essential step. Not only that, the design should be able to deliver ethanol separation economically at the scale considered. Selection of appropriate separation technology, preliminary and subsequent detailed design of an on-farm process, and project economics are the focus of this thesis. Subsequent chapters look at each of these aspects.

The intent of this thesis is to provide documentation necessary to build a demonstration facility capable of producing 10-20 gal/h of 190 proof ethanol. The construction of this state-of-the-art facility will provide evidence for the technological

capability of on-farm ethanol production. Experimentation using the demonstration facility can then be focused at making the process technologically simple enough for farm based operation, low-cost and economically robust. The following research objectives are met in this thesis:

- Design and develop a dewatering process to convert 6-10 wt% ethanol fermentation products to near fuel grade ethanol.
  - Determine appropriate technology for on-farm ethanol dewatering based on a literature survey.
  - Design and simulate the on-farm separation scheme based on representative technical, operating and environmental factors.
  - Determine optimum operating conditions, utility requirements, unit sizes.
  - Conduct an economic analysis for the process. Estimate capital and operating costs for on farm dewatering.
- Finalize the detailed design of the sweet sorghum to ethanol demonstration facility. Detailed design should include
  - Finalize equipment list with quotes.
  - PFD and P&ID of the system
  - Hydraulic analysis of the system.

Chapter II will look into the evolution of the ethanol from sweet sorghum in detail. Chapter III looks at the history behind the concept; when a similar interest in on-farm ethanol production started and why suddenly investigations ended. In chapter IV of the thesis will look at the technological advancements made in the field of separation, how their application to on-farm ethanol production has the potential to change the economics, and the selection process for the right separation technology. Chapter V lays out the design basis of the on-farm ethanol process. The final results capture the detailed design of an on-farm dewatering system to produce near azeotropic 95 wt% (or 190 proof) ethanol from sweet sorghum. This detailed design process including simulation, preliminary design, equipment sizing and economics is discussed in Chapter VI. Chapter VII addresses technical issues related to fouling and feed composition. Conclusions are presented in Chapter VII along with recommendations and future direction of the project.

### CHAPTER II

### EVOLUTION OF BIO-ETHANOL AND SWEET SORGHUM FEED

There has been an alarming increase in emission of green house gases (GHG) over the past years. According to Environmental Protection Agency (EPA) estimates (EPA 2009), in 2007 alone the total GHG emissions were 7,150.1 million metric tons with CO<sub>2</sub> being the primary pollutant. The combustion of fossil fuels was the largest contributor to the production of  $CO_2$  accounting for 80% of the gas in 2007. Even as the debate continues in political circles on climate change and its long term effects, there have been several reports to indicate that the effect of GHGs on climate change is indeed real. In recent years the Intergovernmental Panel for Climate Change, an agency created by the United Nations Environment Programme and the World Meteorological Organization has reported fossil fuels to be one of the main reasons for an increase in  $CO_2$ . The report also states that the phenomenon of global warming is unequivocal and has been corroborated with increasing global average air and sea temperatures (IPCC 2007). These factors, among others, have been the constant driving forces for policy changes and introduction of new "green" policies across the world to help reduce GHG emissions and address the climate change issue.

Petroleum, natural gas and coal account for majority of the fossil fuels. Among the fossil fuels used, liquid fuels account for the largest portion of energy consumption

with 85 million barrels of liquid fuel used in 2006 (EIA 2009), and the Energy Information Administration's (EIA) projections till 2030 account for a steady increase in this trend. The emphasis currently is to bring about a shift from traditional fossil based sources of fuel to more sustainable alternatives. The depleting reserves of fossil fuels pose a threat to its availability. As an example, the ultimately recoverable oil resources have been estimated to be 200-400 billion gigatons (Goldemberg 2000), enough to last only a few generations of populace. Apart from having finite petroleum resources, the extraction of energy is difficult. Only a portion of the oil in the reserve is extracted with primary methods (Dresselhaus et al. 2001). The added problems of political and economic instability created around fossil fuels and the detrimental effects these have on the environment provide the impetus to change over to alternative sources. Renewable liquid transportation fuels or biofuels are important products obtained from biomass. Among several fuels produced ethanol has been the most predominant constituting 99% of all available biofuels in 2005 (Farrell et al. 2006).

### II.1 Renewable Fuels Standard:

The renewable fuels scenario in the United States was not always positive. The production of fuel ethanol in the U.S was negligible before the introduction of federal assistance in 1978. The energy crisis of the early 1970s led to several regulatory measures. Among the very first was the National Energy Act (NEA) of 1978 which was geared towards decreasing U.S dependence on foreign oil by increasing production of domestic energy and the promotion of conservation and efficiency (EIA 2005). In its first year of implementation, 1978, the production of ethanol was below 100 million gal. By

1981, the production of ethanol in the U.S had increased to 175 million gal (EIA 2001). As seen in Fig 2.1, the production of ethanol in the U.S has increased significantly from then.



Fig 2.1: U.S fuel ethanol production and consumption history (EIA, 2008)

The NEA was a significant policy measure which set the stage for alternative energy programs in the U.S. It was followed by several financial incentives introduced by the federal government to boost the production of fuel from renewable sources. These were mainly tax credits and production incentives to partially offset the increased cost (relative to traditional petroleum-based fuels) of producing ethanol. The creation and expansion of markets to encourage and establish renewable fuel production was also initiated. The Energy Policy Act (EPACT) of 1992 introduced the Production Tax Credit (PTC) and the Renewable Energy Production Incentive (REPI) for production of electricity from renewable resources. Several financial incentives for alcohol transportation fuel were established through the EPACT (EIA 2001). These incentives are listed in Table 2.1.

Table 2.1: Federal financial incentives for renew	able transportation fi
Tax Description	¢/gal
Excise tax exemption	5.4
Blenders credit	54
Small ethanol production tax credit	10

fuels

The American Jobs Creation Act of 2004 introduced the Volumetric Ethanol Excise Tax Credit (VEETC) which replaced the blender's credit in place from the EPACT of 1992. Applicable starting January 1<sup>st</sup>, 2009, and continuing until December 31<sup>st</sup>, 2010, the VEETC has a value of 45 ¢/gal which is lower than its original value of 51 ¢/gal. These policies, regulations and financial incentives have spurred the growth of renewable fuels industry. Most of the ethanol produced for transport fuel is blended with gasoline to form E10 or gasohol (ethanol 10% and gasoline 90%) which can be used in cars without engine modifications. Other blends, such as E85 (ethanol 85%, gasoline 15%) and E90, are only gradually gaining popularity with the introduction of Flexible Fuel Vehicles (FFVs) which can use ethanol gasoline blends in any ratio. There are only 1400 E85 fueling stations available to service the 6 million FFVs currently being used across the U.S (RFA 2007). The penetration of E85 in urban markets has been low since most of the fueling stations for E85 are located in the Mid-west.

Fuel grade ethanol, used for blending into gasoline to form E10 or E85 blends, corresponds to 99+ vol% ethanol content. From several specifications available for 'fuel grade ethanol' two are listed in Table 2.2.

Component	Measuring unit	Magellan	ASTM	Limits
Ethanol	vol %	93.500	92.100	Min
Methanol	vol %	0.500	0.500	Max
Water	mass %	0.820	1.000	Max
Sulfur	ppm	10.000	30.000	Max
Solvent washed gum	mg/100 mL	5.000	5.000	Max
Sulfate	ppm	4.000	4.000	Max
Chloride	mg/mL	32.000	40.000	Max
Copper	mg/mL	0.080	0.100	Max
acetic acid	mass%	0.007	0.007	Max
Denaturant	vol %	1.960	1.960	Min
Denaturant	vol %	5.000	5.000	Max

Table 2.2: ASTM and Magellan Pipeline specifications for ethanol

There are several standards available for fuel ethanol and these differ with participating agencies. For example, pipeline carrying ethanol demand a slightly more stringent specification to cover for any impurities or water pick up during transfer described in detail in Table 1.2. As seen in Table 1.2, the minimum ethanol content is different in the fuel ethanol specifications published by Magellan pipeline and ASTM specifications at 92.1 vol%. Anhydrous ethanol (containing <1% methanol, water) obtained from manufacturing units is diluted with denaturant to give rise to the final fuel concentrations.

Currently, this fuel grade ethanol is obtained predominantly from corn in the U.S. In the transportation fuels sector alone, by January 2008 the U.S had 139 biorefineries online using corn as feedstock capable of producing 7.8 billion gallon of ethanol annually (RFA 2009). Highlighted earlier, this feedstock has run into a host of problems and controversies over its energy balance, effect on food and animal feed prices and ecological impact. Several alternative feedstocks are now being looked at for manufacture of ethanol. One of the many sugar based crops (which include sugarcane and sugarbeets) is sweet sorghum.

#### II.2 Sweet Sorghum as Ethanol Feedstock:

Sweet sorghum provides many sources of energy. The grains house starch and the sugars are stored in the stem of the plant. The main source of carbohydrate, sucrose, is present mostly in the stalk with varying amounts of starch or reducing sugars. The leaves and the dried stem constitute the baggasse and are often used as boiler fuel (Saballos 2008). All three portions; the grains, the stalk, and the leaves of the plant in some way can be utilized for energy production. This factor makes sweet sorghum a very lucrative

energy crop. Historically it has been used to derive grains and sugars for food including beverages and forage for livestock.

Sweet sorghum has a higher net energy yield per gallon ethanol produced when compared to corn. The net energy gain from sweet sorghum has been estimated to be 22,000 Btu/gal (Meo et al. 1982); higher than corn (the net energy output was evaluated at -5,645 Btu/gal) and one of the highest amongst several crops analyzed, including wheat (4,425 Btu/gal). A major reason behind this is that the production of ethanol from sweet sorghum is direct compared to starch and cellulosic feedstock as the carbohydrates produced by sweet sorghum are sugars which can be directly fermented to ethanol. It also has a high yield: 300 to 600 gallons of ethanol per acre (Stotts 2007), lower water consumption, and requires less fertilizer. The water requirement for sweet sorghum is a third of that for sugarcane and half of that required for corn (Grassi 2005). Ethanol production from sweet sorghum also has the advantage of producing lower amount of effluents. It produces 3 L effluents/L ethanol produced which is six times less when compared to grain crops (Palaniswamy 2007). The phrase "more crop per drop" is thus often used for sweet sorghum in context of these advantages (Weller 2007). Additionally, ethanol obtained from sweet sorghum contains less sulfur and has a higher octane number compared to other sugar based crops (Reddy 2007). These factors provide a strong driving force to use sweet sorghum as a source for fuel ethanol.

#### II.3 On-Farm Sweet Sorghum Bioethanol:

Sweet sorghum was considered as a front runner energy crop even in the early 1980s (Meo et al. 1982). As a sugar based energy crop, sweet sorghum was identified as

a potential long term solution in DOE sponsored studies (Nathan 1978). Evaluations carried out on sweet sorghum for small scale ethanol producing plants gave positive results for the crop in terms of ethanol yield and conversion efficiency. The theoretical average ethanol yield was calculated to be above 400 gal/acre of sweet sorghum (Bryan et al. 1981). However, several barriers were identified at that time. Acidification and subsequent loss of ethanol and low recoveries of sugar from the stalk after pressing the stalks were the main problems faced in the process. The authors suggested multiple passes of the cut stalks through the press for better juice extraction. Other studies had similar problems with the harvest of the crop and storage of the cut stalks and pressed juice (Demmel 1981). Cut stalks stored for close to five months gave only 15% the amount of juice fresh pressed stalks had given. A large portion of the sugar contained in the stalks had gone into dry state and were not in extractable form. The presence of leaves and its removal posed difficulties in harvesting sweet sorghum stalks for the Demmel team.

The issues limiting current applications of sweet sorghum are found to be very similar. Seasonal operation of sweet sorghum based plants, fermentation and ensilage of the harvested crops pose major barriers (Bellmer et al. 2007). A low cost in-field ethanol production facility is one way to circumvent these problems. One needs to ferment the pressed juice on-farm immediately to avoid the conversion of sugars into products other than ethanol. For more efficient extraction of juice from the stalks a new approach has been devised.

Harvesting and pressing the sweet sorghum stocks involves operation with a tobe-patented prototype harvester, as seen in Fig 2.2, and the subsequent in-field

fermentation of the pressed juice (Kundiyana 2006). The in-field harvester has a forage chopper head and built-in juice rollers. The stalks are cut, the juice is pressed as they pass through the rollers, and are subsequently left behind on the field as 'baggasse.' The pressed juice is pumped and collected into a bladder or storage unit which moves along with the harvester. The harvesting and juice collection is achieved in a single pass.



Fig 2.2: In-field harvest and sweet sorghum juice collection (www.kitchensink.okstate.edu)

Smaller scale units have several technical factors working in their favor.

Transportation of water rich juice extracted from sweet sorghum is not an issue since all subsequent dewatering activities are on farm. Wastes may be used on farm as fertilizers or animal feed. The farmer gets to keep a larger share of the income. A study done in the department of Agricultural Economics at OSU simulated the economics of producing ethanol from sweet sorghum on farm to. The net present value for a sweet sorghum based ethanol on-farm ethanol facility, with a 500 acre planted crop area, operated over ten years, was calculated to be \$2,714,867 (Fryer 2008). The average rate of return was calculated to be 47% from several simulated cases. A large portion of the capital investment was attributed to the investment in processing equipment such as bladders for

fermentation, distillation unit and press for juice extraction. With the participation from farmer co-operative organizations and joint investments, these costs can be shared. The use of sweet sorghum as a potential feedstock for a small scale farm set up has many promises. While studies of on-farm ethanol show positive signs of development, simultaneously ethanol production from sweet sorghum is gaining popularity across the world in large scale processing scenarios as well.

### II.4 Industrial Demonstrations of Ethanol from Sweet Sorghum

There has been a lot of interest in ethanol production from sweet sorghum on a larger scale in the U.S and world over. In the United States, Texas Bioenergy Marketing Associates plan to build five farmer co-operatives, each having a capacity of producing 12 million gal/year ethanol (Emison 2007). The state of Florida has also seen activity in this field. Renergie Inc. has received a 1.5 million dollar grant to design and build a small scale ethanol plant based on sweet sorghum (Lane 2008). Sweet sorghum is being actively investigated outside the U.S as well. International Crops Research Institute for Semi Arid Tropics (ICRISAT) in a partnership with Rusni Distilleries has set up a 11,000 gal ethanol product/day plant in India. This prototype plant produces fuel grade ethanol using molecular sieves (Reddy 2007). China and the Philippines are also working to establish sweet sorghum based plants. The Ministry of Agriculture in China along with the Food and Agricultural Organization of the UN plans to set up ethanol manufacturing plants in Shandong and Shaanxi provinces (FAO 2002). In Philippines, state owned Philippines National Oil Corporation has reached an agreement with UK based NRG Chemical to set up biorefineries using sweet sorghum in the near future (Saballos 2008).

### CHAPTER III

### PREVIOUS INVESTIGATIONS IN ON-FARM ETHANOL

Several projects were initiated by the Department of Energy (DOE) and United States Department for Agriculture (USDA) in the late 1970s and early 1980s. This was primarily in the wake of the energy crisis faced by the United States in the 1970s, which saw skyrocketing oil prices. Crude prices tripled and doubled in 1973 and 1979 due to oil embargos and controlled oil production by the Organization of Petroleum Exporters (OPEC) (Campbell et al. 1998). There was an increased interest in several renewable technologies including solar, biomass and wind. Many projects received funding from DOE. As clearly seen from Fig 3.1 the funding for research and development work for renewable energy received a major boost between the years 1978 and 1983.



Fig 3.1: R&D funding from selected renewable technology Source: EIA, Renewable Energy technology 2000: Issues and Trends (1999 USD)

Projects identified by DOE at the time were aimed at fulfilling the immediate energy needs for the country and the focus was thus on corn ethanol, which was believed to have the potential to realize those needs. These projects often had a "quick-fix approach" intended to meet the goals as quickly as possible and thus were not technologically efficient or cost effective (Wyman 2001). There was specifically a large amount of interest in the Mid-Western states which grew much of the corn and housed a significant portion of the agricultural population (Hohmann 1980).

Early efforts even included a look at the possible role of the beverage distillery industry in the production of alcohol for fuel (White 1979). Since the technology for alcohol production was already set up this was thought to be justified extension to meet the growing demand of ethanol. At the time the number of plants capable of manufacturing fuel ethanol was found to be too low as only three had the capability to produce 200 proof ethanol. The degree of variation was found to be too high for the industry to provide a single fuel product and thus not much activity was seen in this regard thereafter. There were projects which were also looking at the use of geothermal energy incorporated into the production of alcohol (May et al. 1979). Apart from these early ventures, there were several major projects initiated at the time, sponsored in part by the DOE and USDA involving the construction of farm scale pilot facilities at major universities. These were built and operated to test the feasibility of on-farm production of alcohol from corn feedstock. III.1 Investigations on Bioethanol in Universities across United States:

One of the projects was undertaken at the South Dakota State University (SDSU). A multidisciplinary team comprised of mechanical and agricultural engineers, agricultural economists, dairy scientists and microbiologists undertook the task of producing ethanol on a small-scale or "on-farm" system (Westby et al. 1982). The focus of their study was the production of 190 proof ethanol using corn as feedstock. The study was conducted from 1979 to 1983 and a fully operational unit was built on the SDSU campus. The distillation unit for this process was manufactured by Arlon Industries, had stainless steel exterior, and a fiberglass interior. There were two columns, each with a height of 4.33 m, and an ID of 0.305 m. The stripping column had 15 trays which were spaced at 27.9 cms, and the rectifying column had 23 sieve trays spaced 14 cm apart. Overall, the unit was capable of producing 65 gallons ethanol/week. A photograph of the unit is shown in Fig 3.2 (b). From the photograph one can make out the two columns distinctly.

An engineering economic study based on the process was also undertaken to establish the feasibility of the venture (Dobbs et al. 1984). The best case scenario where a 175,000 gallon ethanol/year farm scale plant had a readily available market for azeotropic ethanol was evaluated as barely being able to break even and it was concluded that the "prospects of plant feasibility seem poor." The study determined the lack of a definitive market for wet alcohol and grains along with the cost of the transportation of the products to their desired destinations as major factors for unfavorable economics.



Fig 3.2: (a)Distillation apparatus used at University of Alabama (Eley et al. 1983)and (b) at SDSU (Westby et al. 1982)

To reduce the then high cost of producing ethanol, several modifications to the unit were introduced into the process: recycling stillage, supplementing corn feedstock with cheese whey, and increasing levels of starch in the mash (Gibbons et al. 1983). The team looked at the possibility of using fodder beets as an alternative feedstock in conjunction with a continuous solid state fermentation system (William R. Gibbons 1984). An energy balance for the distillation portion of this plant suggested that the size of the distillation operation and the reflux ratio were key factors in determining energy requirements. The expenditure of energy by the distillation operation was 3.2 - 5.0 MJ/L ethanol, comparable to other farm units set up at the time (Stampe et al. 1983). Suggestions made to reduce the expenditure of energy included:

- a) Adding a heat exchanger between the stripping and the rectifying column to extract heat from the stripping column products
- b) Recycle excess heat from condenser to increase temperature of fermentation contents.

- c) Reduction of thermal losses by adding insulation on the distillation columns
- d) Using the correct number of plates for the distillation columns.

Another undertaking was started at University of Alabama, Hunstville in conjunction with the Madison County Farm Bereau as part of the Fuel Alcohol program and was funded by DOE (Adcock et al. 1981). A small scale unit designed for continuous production of ethanol was installed and operated at the university. The distillation unit at this facility was built by Industrial Innovators Inc. and constituted a typical farm scale set up. There were two columns, the beer column with 18 trays and the stripping column with 27 trays. Corn feedstock was used here as well. The output of the plant, 10-15 gal/h of 170-190 proof ethanol, was achieved only after several technological difficulties had been eliminated. These included loss of ethanol through the stillage, leaks in the columns, pump cavitation and column flooding (Eley et al. 1983). The optimum feed flowrate for the plant was 140 gal/h at which the ethanol product generated was between 9 to 14 gal/h. Key features of the plant which set it aside were use of a rotary screw press to deliver the feed to the distillation columns and the use of polyolefin storage tanks for the feed and the product. To increase the productivity of the plant the team later introduced the use of packing into the rectifying column. Even with these changes the plant produced only 14 gal/h 170 proof ethanol. Though the change was able to modify the economics and make the process of producing alcohol a little cheaper, several technological issues still persisted, which resulted in heavy losses of ethanol (Adcock et al. 1982).

Investigations regarding the production of on-farm ethanol was simultaneously being undertaken at Iowa State University where a small scale distillery had been set up (Ozkan et al. 1981). The plant included tanks, condenser, heat exchanger,

pumps and a distillation column. Researchers at Mississippi State University were investigating the construction of the distillation column from plastic. They set up a 6 inch ID column made from a section of a PVC schedule 80 pipe (Lightsey et al. 1982). The column managed to produce 188 proof alcohol but not without losing some of its structural integrity. Due to a bend along the height of the column it had to be supported externally with the help of wooden planks to ensure smooth operation.

Though several universities were involved with research on setting up an on-farm ethanol production process, the design, selection and manufacture of ethanol separation and dewatering unit operations were often outsourced to private still manufacturing companies. Most of the pilot scale ethanol separations units in various universities were packaged units bought from still manufacturing companies on order. Several enthusiastic farmers also purchased these alcohol stills hoping to become energy independent in a short time. A wide variation in the type of stills was seen due to a lack of standardization. Some were not even able to meet the claims made by the manufacturers. Attempts were made to set procedures which would, in an unbiased manner, compare varying still designs on their energy requirements and production rates (Rein et al. 1983). However a statute or law regarding the manufacture of small scale alcohol was never put in place as the concept did not take off successfully.

About the same time period an on-farm ethanol manufacturing pilot plant was set up at Clemson University (Dodd et al. 1981). The pilot plant was specifically designed with on-farm operating conditions in mind. The main focus of the project was to optimize costs and energy expenditure associated with the process without making the process labor intensive. A packed bed distillation column was connected to an integrated

batch cooker-fermentation tank, as seen in Fig 3.3, and the product output was 8 gal/h of 180 proof ethanol. The unit was used to study ethanol production from corn and other sugar based crops such as sugar beets and sweet sorghum. Apart from adopting a batch operation what set this study apart from other attempts was the fact that

- a) The facility was made mobile by setting it up on a 44'x8' trailer truck.
- b) Ethanol proof testing device was installed in-line at the product and bottom discharge.

Several demonstrations were also given as part of their outreach program.

However, like other on-farm projects this study was also discontinued.



Fig: 3.3: Process Flow Diagram for On-farm ethanol plant at Clemson Univ. South Carolina (Dodd et al. 1981)

Activity was also seen at the Illinois State University (ISU) and Ohio State University (Longbrake 1983). At ISU, a small unit was set up to produce 180 proof ethanol (ISU 1982). The unit produced 160 gal/week enough to supply campus wide research activities, such as engine modification projects, with fuel alcohol. The batch still unit was an off the shelf packaged unit bought from Tri-Star Corporation. The project generated significant enthusiasm in the local community but was not able to operate economically.

Many of these studies suffered from being highly labor intensive. Manual temperature control for the column reflux and steam rate often resulted in off-spec products and could have resulted in un-safe conditions. Complete automation was often not incorporated due to high costs which would make the on farm unit undesirable to farmers. To offset at least some of the costs for labor partially by automation, a team of agricultural engineers from the University of Nebraska undertook a project to study this possibility. They identified column beer feed rate and steam input as the key variables to be controlled. Condenser cooling water, liquid levels, column and temperature and pressure were identified as the other variables for possible control utilization (Sullivan et al. 1983). A detailed pilot plant operation to study the cost implications was planned for but not put into place.

More often than not the projects started would often look at ethanol-water separation as part of the bigger problem of making on-farm ethanol a reality. The approach was to purchase the separation unit from external vendors and fit it into the larger scheme of the biofuel production process. This approach as seen from several examples did not reap large benefits. It was the ethanol water separation or dewatering portion of the process which caused the largest number of technical difficulties and the teams were often ill equipped with technical know-how and tools to tackle them. The economics and feasibility of the ethanol process, based on these cases, was then judged to be impractical, which seems to be unfair towards the success of the on-farm concept. It is

possible then that the ethanol dewatering process, if designed to be technologically adept and advanced, can be economically feasible.

III.2 Independent Investigation and other Small Scale Studies:

Breaking away from studies conducted by universities all over United States, there were several other smaller DOE funded initiatives which were keenly taken up by independent investigators and farmers. Their efforts were mainly dedicated towards finding short term solutions and included the design, fabrication and operation of batch type prototype stills, as shown in Fig 3.4 a (Lowe 1980; Demmel 1981; Hegeman 1981). These types of units often had a single tank or chamber for fermentation and cooking along with either one or two columns attached directly above it for distillation. In some cases these units were made mobile by mounting on trailers as seen in Fig 3.4 c (Glenn 1983). There were also several grants from the DOE to investigate the feasibility of using solar energy during the process of ethanol production. Several of these small scale studies incorporated reflectors or equipment to harness energy for the sun to evaporate ethanol and can be seen in Fig 3.4 b (AAF 1981; Fitzcharles 1982; MCCSI 1983; Eyrich 1984; Kriley 1985).

In small operations described previously, the production of ethanol ranged from 10-20 gal/h with high variability in the product concentration. Final product concentrations ranged anywhere from 160 to 190 proof ethanol. Production of ethanol was highly labor intensive and inundated with technical glitches. Pipeline clogging, pump plugging, temperature variation, off specification products, loss of alcohol, and materials handling were identified as some of the problems.



Fig 3.4: Small projects for on farm ethanol production: (a) Ark-o-hol community experiment group (Lowe 1980) (b) Solar powered still (Fitzcharles 1982) (c) Schematic of portable ethanol production facility (Grinnell et al. 1986)

Based on the events which were taking place in the agricultural community, several how-to-do-it "manuals" were developed which compiled the attempts of these farmers involved in making alcohol on the farm (Nellis et al. 1979; Carley 1981). In the early 1980s the Solar Energy Research Institute (SERI) also came out with a practical users' guide which was meant to assist farmers with the technical details of constructing a fuel alcohol plant (Tarr 1982). The main intent was to make technical information more accessible to an average farmer.

There were many other small experiments which the DOE showed interest in and funded for a brief period of time. Several other projects explored around the time included dehydration of alcohol by using a vacuum shell (Agri Stills of America 1981). Inventors were also able to make and patent their own alcohol making stills, which were mainly based on the "moonshine" concept (Iowa Farmers Union 1981). These activities
were for the most part extremely scattered and a by-product of a strong desire in the agriculturalists to experiment their way to fuel independence.

Several other attempts at continuous processing were also more or less unsuccessful. The units were fabricated and constructed with much enthusiasm and optimism but due to lack of technical experience, the inventors who set up and operated these small scale plants ran into a host of problems and operational glitches (Sweany 1982). In contrast, the outlook was often upbeat in some of the participants who found the venture had "simple and proven technology" (Hall 1981). There were others who thought they "lacked technical information and technically qualified people" (Helstad 1981). Government documents intended to shape policies and inform farmers of their options related to small scale ethanol production suggested that smaller plants might not have a competitive cost advantage over larger facilities (Kerstetter 1984). While some efforts looked promising (Jantzen et al. 1980; Pile et al. 1981) most of the efforts, which were part of the experimental small scale Alcohol Fuels Project funded by DOE or other farm development projects, ran out of steam by the mid 1980s as the operation of these investigative small scale plants was found to be un-economical.

#### **III.3** Feasibility Studies

The only shift from this approach of setting up experimental ethanol producing facilities was seen in a techno-economic feasibility study undertaken by Raphael Katzen Associates International Inc., Ohio (Raphael Katzen Associates International 1980) for the U.S National Alcohol Fuels Commission. The goal of their evaluation was to optimize the costs and investments for ethanol production processes while making sure

the plant would be easily operable by on-farm or co-operative personnel. Based on process designs for ethanol production capabilities from15 to 150 gal/h, the evaluations were done for both 190 and 200 proof products. The approach was meticulous and detailed. An important feature was the inclusion of fusel draws in their process flow diagrams. They acknowledged the instability that the heavier alcohols would bring to the distillation system and incorporated draw offs at appropriate locations. Their results suggested the operation of a 15 gal/h ethanol producing plant would prove to be unprofitable. Their recommendations were geared towards the construction and operation of a 150 gal/h plant owned as a co-operative.

A similar design exercise was undertaken by the Department of Chemical Engineering at the University of Oklahoma. Their analysis ranged for plants producing 180-190 proof ethanol at 10-50 gal/h. The study incorporated a two column distillation set up with 27 actual plates for the stripping column and 40 trays for the rectifier. This was perhaps the most detailed design that was proposed at the time (Radovich et al. 1981). Key features and suggestions of the design are listed below.

- a) Inclusion of fusel oil draws in the rectifier where the alcohol concentration would be 130 proof.
- b) The incorporation of molecular sieve dehydration as an economical option
- c) Suggestions regarding the use of reboilers as against steam to reduce waste production and energy usage
- d) Suggestion of incorporating air cooled heat exchangers for condensers
- e) A strong focus on safety
- f) Use of simulations software for distillation column design

The economic evaluation by OU was supposed to be conducted based on actual small scale plant data. However, the interest in small scale ethanol research was waning and few takers were at hand to participate in an economic evaluation. This was one of the few detailed technical studies on small scale ethanol processing.

The involvement of chemical engineers in most projects was minimal and it is not surprising thus that the separation techniques employed were rudimentary or off the shelf. In 1983, White issued a report on behalf of the Department of Energy that reviewed the status of small, farmer-operated ethanol production facilities (White 1983). He noted that many of the problems with the on-farm facilities operating in 1981 were attributable to "poor technical advice and inadequately-proven plant designs." Interest in fuel ethanol production was waning by this time due to a significant decline in oil prices and the resulting loss of state gasohol exemptions. White's report noted that "the distillation columns and associated equipment represent a major fraction of plant capital costs and consume a large portion of plant energy." These factors combined with the small scale of the on-farm operation presented significant economic obstacles that remain even today.

Economic studies of small scale farm or cooperative owned plants more often than not conclude that the size was against the operation (Farmer Co-operatives 1981). Lack of market for azeotropic ethanol, by product recovery, primitive technology and high costs were some of the major reasons cited (Atwood et al. 1980; Iowa Cental Community College 1982; Bowker et al. 1983). Even those analyses which projected the small scale alcohol ventures to be profitable cautioned against the sensitivity of the economics to various factors such as labor, feed costs and availability and actual ethanol yields (Schafer 1980). There was need at the time to build pilot plants which would

efficiently produce ethanol and would be able to shed some light on the techno-economic questions related to the operation. The approach however would have to be different from the approach of the earlier "quick-fix" attempts where importance would be given to detailed engineering design of the process, on the lines of Katzen or OU's study. The work started in the late 1970's and early 1980's for on-farm distillation was never completed. Initial results (White, 1983) revealed the need for improvements to reduce costs but the required follow-up was never initiated due to the rapid decline in oil prices in 1982.

III.4 More Recent Small Scale Ethanol Facilities

To prove the success of any ethanol producing facility there is a need for demonstration units to guarantee process feasibility and engineering success. One such demonstration facility has been set up in the recent past by National Renewable Energy Laboratory (NREL) and DOE. The Process Development Unit (PDU) is equipped with state of the art technology and generates process data necessary for scale up and process feasibility studies (Schell et al. 2004).



Fig 3.5: NREL Biomass Process Development Unit

The PDU has generated important results regarding the use of various feedstocks, corn fiber being one of them. The pilot plant distillation system employs a direct steam injection tower which has a couple of pre-heaters preceding it. The tower has 19 sieve trays, is 32 ft tall and 1.3 ft in diameter and operates for 5-6 hours a day. For operation on a smaller scale, NREL has a mini-pilot plant to test biomass pretreatment options and fermentation processes. Distillation or ethanol water separation processes is not a focus of these units (NREL 2000). Even though the PDU processes a fairly large quantity of feedstock (1 ton per day), much larger than a small scale on-farm plant, it highlights the importance of setting up a demonstration facility.

The on-farm ethanol dewatering demonstration facility at OSU aims to incorporate learning from previous efforts into the design and demonstrate that ethanol manufacturing on a small scale is not only technically feasible but economically sensible. Experimentation on the state-of-the-art demonstration facility will put to rest questions concerning economics and technical feasibility, bringing the on-farm ethanol concept closer to reality.

# CHAPTER IV

# TECHNOLOGICAL ADVANCEMENTS IN DISTILLATION

Over the past two decades several technological advances have taken place pertaining to the field of distillation. More efficient tray designs are now available based on an improved understanding of tray hydraulics, mass transfer and heat transfer (Jose et al. 2000). Column internals are custom designed for fouling services such as ethanol dewatering and draw very strongly on the hydraulics, mass transfer and separation mechanisms of the process (Sinderen et al. 2006).

### **IV.1** Column Internals for Bioethanol Processes

For the ethanol separation process which deals with much of the solids contained in the feed, it is essential that the column internals are capable of handling high levels of fouling. For this reason, historically, there have been two columns present in the production of bio ethanol among which the first column is equipped with trays. Due to the large amount of solids, protein matter and sugars associated with the feed, packing is avoided in the first column and the installed trays need to be able to avoid solid build up to maximize plant efficiency. Some of these trays used in industrial scale bioethanol plants are specific to fouling services and are encountered frequently in production (Pham et al. 1997; Daniel R. Summers 2006). Among them, the V Grid trays have become extremely powerful in anti-fouling applications. Developed first in the early 1960s by Nutter Engineering, V-grid trays were then modified through to the 1990s and are today available in Mini, Small and Large sizes (Chemtech 2008). These trays utilize a combination of features from sieve trays and floating valve trays. There are fixed projections from the surface of the trays which act like fixed valves. The openings are oriented along the flow of fluid. The smaller the size of the openings the higher the capacities of the tray due to reduced pressure drop. An example of a V-Grid tray can be seen in Fig 4.1



Fig 4.1: V-Grid anti fouling trays (Sinderen et al. 2006)

These trays offer a marked improvement in turndown and have anti-fouling capabilities (Summers et al. 2001) which make them appropriate for use in the sweet sorghum bioethanol application. Trays are compared on several criteria, efficiency, pressure drop, and turndown ratios being some factors. Typical sieve tray turndown ratios (maximum to minimum vapor capacity of tray) are usually found close to 2:1 (Kister 1992) and the V-grid trays demonstrate a far better turndown close to 5:1. A modification to the V-grid tray applied at an industrial corn ethanol plant was able to deliver 61% efficiency which was a large jump from the 48% efficiency the plant was experiencing previously (Summers et al. 2002).

In addition to more efficient trays, the use of packing for separation and purification provides an opportunity to increase the overall efficiency of the process. In early investigations of on-farm dewatering, the rectifier was operated with trays. Random packing was tested to improve column performance and an increase in column efficiency was seen (Eley et al. 1983). Now, the introduction of structured packing offers an opportunity to increase separation efficiencies even further. Structured packing has 50% more open area than random packing providing it higher capacity. In this type of packing, the wetted area provided is double that found in random packing which aides it in achieving high mass transfer rates and thus increased separation efficiency (Lieberman et al. 2008). In most flow ranges the structured packing efficiency will be 50% or higher than the either random packing or trays (John J. McKetta 1997). Another concept used to determine column performance is Height Equivalent to Theoretical Plate (HETP). These values are generally obtained through extensive experimentation on lab or large scale columns (Seader et al. 2006). HETP is the ratio of the packing height to the number of equivalent equilibrium stages. A low value of HETP is desirable as it indicates the use of lesser packing with higher efficiencies. From Fig 4.2 we see that



Fig 4.2: HETP vs. specific surface area of metal packing. System: Chlorobenzeneethyl benzene, 50 mmHg at total reflux (Kister 1992)

structured packing has a lower HETP (<10 inch) than random packing (pall rings, 25-30 inch). Thus the use of structured packing in part of the process will reduce cost of the rectifier column.

**IV.2 Design Improvements:** 

Greater understanding of the ethanol-water system, design and simulation capability and process integration opportunities are the other advantages for ethanol water design twenty years after the earlier attempts. Early design methods for distillation columns were based on graphical two-dimensional McCabe-Thiele diagrams. However with the advent of computers and the use of process simulation software like CHEMCAD, ASPEN or HYSYS there has been a vast improvement in the flexibility of the design process and now large number of compounds can be included in the design with greater accuracy (Barnicki et al. 2004).

Some other factors which could play a role in making the on-farm process economical are

- a) Improved monitoring and control capabilities. A higher level of control will lead to lower losses of ethanol through effluent streams resulting in higher yield.
- b) Improved heat exchanger design and fabrication techniques will ensure minimum energy wastage and increased energy efficiency of the demonstration facility.
  Improving the efficiency of equipment used in the process will reduce the capital cost (due to the smaller size) while improving economics (due to the higher yield).

These factors provide us with a driving force to reinvestigate on-farm ethanol separation. The result of this re-examination will establish the appropriate technology for on-farm ethanol production.

IV.3 Selection of Appropriate On-Farm Bioethanol Production Technology

Complete removal of water from ethanol or vice-versa is complicated by the presence of an azeotrope at 95 vol% ethanol – water solution. Distillation provides economical separation of ethanol till the azeotropic concentration. Beyond this point a different technology is needed to replace distillation. One reason is the energy and the cost of distillation increases exponentially with the purity of ethanol in the product, and thus simple distillation cannot be used beyond the azeotrope for dewatering to produce anhydrous or fuel grade ethanol economically (Leeper 1992). This is shown in fig 4.3 (a). The energy required to produce anhydrous ethanol reaches 3000 Btu/lb, three times the energy requirements to produce azeotropic ethanol.



(a) (b)
 Fig 4.3:Energy requirements for distillation (a)Energy requirements of fractional distillation as a function of distillate mass fraction (b) Energy requirements for fractional distillation as a function of feed concentration (Leeper 1992)

To reach fuel grade ethanol it is thus beneficial to use a combination of technologies: distillation to obtain azeotropic ethanol at 95 wt% and a subsequent dewatering step to obtain >99 wt% ethanol. This concept is widely used in industrial ethanol applications where distillation combined with a dewatering step is commonly used.

Several technologies are available for dewatering, such as azeotropic distillation, solvent extraction, super-critical fluid extraction (Seader et al. 2005), salt-extraction (Furter 1993), gas stripping (Cen 1993), reverse osmosis (Choudhury 1985), dialysis (Ladisch et al. 1979) molecular sieve adsorption (Huang et al. 2008) and membrane based technologies such as pervaporation (Slater 1989) and vapor permeation (Sander et al. 1991). Each of these technologies has been successfully applied to ethanol dewatering applications at an industrial level. There are other technologies which are promising but still in the research stages such as bio adsorption (Ladisch et al. 1979), perstraction (Grobben et al. 1993) and liquid membranes (Belafi-Bako 1995).

Distillation is a capital intensive, high energy process which facilitates separation of a mixture. In large scale applications such as for petroleum or chemical plants, this cost may be greatly reduced to the order of just pennies per gallon as the high fixed capital costs are spread over a large quantity of product. This cost becomes significant when the throughput becomes small as in the case of an on-farm ethanol dewatering system ( $\leq 25$  gal/h of ethanol product). Unfortunately, none of the separation technologies listed previously to provide 95 vol% ethanol provides a more economic alternative to distillation at on-farm throughput rates at this point in time.

There is a strong driving force to provide the final dewatering step on farm as well. Producing fuel grade ethanol would increase the economic benefits for a farmer. Along with personal economic growth this gives rural development a big boost by assisting agricultural producers to become fuel suppliers. Since distillation is uneconomical for dewatering due the exponential increase in energy, as mentioned earlier, there is a need to establish alternatives which can be used for on-farm ethanol dewatering. Key features on which further selection can be made are:

- a) Mechanical and operational simplicity
- b) Ease of maintenance
- c) Substantial water removal and minimal ethanol loss
- d) Costs
- e) Energy expenditure

Each of these features will be assessed in the following sections in the context of on-farm ethanol dewatering.

### Mechanical and operational ease:

This would translate to complexity in design, number of units required in the process and the kind of utilities involved. Table 4.1 highlights some of the disadvantages and advantages of ethanol separation technologies. For example both extractive distillation and azeotropic distillation require one or two additional towers respectively (Huang et al. 2008). On the other hand molecular sieve adsorption operation requires packed columns which are smaller in height and more efficient and a more desirable option. A molecular sieve design specific to OSU's on-farm ethanol dewatering needs was carried out by UOP, a leading company in membrane technology. A total of two

sieve beds, each 14 ft in height is required for the final dewatering step (190 to 199+ proof). This corresponds to a total of 7200 lbs of 3A-AG, 1/8 inch molecular sieve material. The packing material is often in the form of granules which are commonly zeolite.

Separation technology Key aspect		Advantages	Disadvantages		
Azeotropic Distillation	Addition of solvent	Highly selective solvents present	Health and safety issues. Benzene - carcinogenic cyclohexane - flammable		
		Complete recovery of ethanol possible	High energy and cost: recovery of solvent		
Extractive		Bio-compatible solvents present: Dodecanol	High solvent needs: Solvent-feed ratio = 1		
Distillation		Complete recovery of ethanol possible	High energy and cost: recovery of solvent		
			Corrosion of equipment due to salt.		
		Recovery of ethanol high	Equipment needs to be made of more		
Saline extraction			expensive material.		
			Solids handling difficult		
		Prefabricated membrane modules	High membrane cost		
Pervaporation	Membrane based	Lower energy usage	Membranes need to be more selective to ethanol		
Vapor permeation		No material handling problems	Fouling issues		
		Works well with low ethanol			
Reverse Osmosis	-	concentrations (1 - 3 wt%)			
Adsorption	Adsorption by solids	High ethanol recovery	Regeneration of adsorbent: High steam requirement		
		Lower costs	Automation scheme: complicated		

Table 4.1: Qualitative comparison of ethanol dewatering technologies

Bio-based starch and cellulosic materials such as corn cobs, corn meal, wood chips, bagasse have been experimentally investigated for ethanol dewatering capabilities(A. Ostroff 1988). Membrane based operations such as pervaporation and vapor permeation are both available in modular units which can be attached to the distillation column (Roza et al. 2006). The uses of solvents in azeotropic and extractive distillation pose some problems. The most common solvent used in azeotropic distillation is cyclohexane. However, due to its flammable nature special precautions must be taken into consideration for storage and operation. Bio-compatible solvents such as dodecanol are being considered, however the emphasis is on use in integrated fermentation separation operations (Huang et al. 2008).

#### Ease of maintenance:

For an on-farm separation application the frequency and simplicity of maintenance become an important consideration. Not only should the technology in question require as little maintenance as is possible, the technology should be simple enough for farm based operation. From a maintenance standpoint, salt extraction has several disadvantages. Salt based operations are susceptible to corrosion (Pinto et al. 2000) and require expensive solid handling equipment and special care. Processes which deal with addition of solvents have more equipment, separators or columns which cause additional maintenance issues.

# Ethanol recovery and loss:

Solvent based recovery or dehydrating technologies have extremely high recovery and are able to recover all the ethanol present in the process (Black 1980). Information on recovery efficiencies of membrane based and adsorption processes are not readily

available. Table 4.2 highlights some of these values. However for a substantially concentrated feed stream containing > 90 wt% ethanol, the final product is fuel grade ethanol i.e. > 99 wt%. Vapor permeation can also be used to dewater ethanol streams containing as low as 70 wt% ethanol (Sander et al. 1991).

Cost <sup>2</sup> (USD/lb)	Energy Requirement <sup>1</sup> (GJ/m <sup>3</sup> ethanol)	Ethanol recovery <sup>3</sup> %	Final Product (wt%) <sup>1,2,4</sup>	Ethanol Feed (wt%) <sup>1,2,4</sup>	Separation Method
		,			Conventional
	6.7 - 8.2		92.4 - 95.6	7 - 10	Distillation
3.65	0.40		99.9	95.0	Pervaporation
	0.64		10.0	4.0	Reverse Osmosis
3.24			99.5 - 99.9	70 - 94	Vapor permeation
				92.4 -	Azeotropic
4.31 - 3.04	1.9 - 2.0	100	99.9	95.0	distillation
	· · ·	· ·		· · ·	Extractive
		100	99.8		distillation
		100	99.8	5 -7.5	Saline distillation
					Adsorption
4.27			> 99.5	> 90	/Molecular sieve

Table 4.2: Comparison of ethanol dewatering technologies

1. Li, N. N. and J. M. Calo (1992). Separation and purification technology, CRC Press.

2. Sander, U. and H. Janssen (1991). "Industrial application of vapor permeation." Journal of Membrane Science 61: 113-129.

3. Black, C. (1980). "Distillation modeling of ethanol recovery and dehydration processes for ethanol and gasohol." Chemical engineering progress 76(9).

4. Seader, J. D. and E. J. Henley (2005). Separation Process Principles.

Reverse osmosis can be used to concentrate ethanol from a water rich stream and

this process is often used in alcoholic beverage processing industries (Lea et al. 1995).

Reverse osmosis applications at high ethanol concentration have not been as popular. Gas stripping is also used to recover ethanol from fermentation broth feeds and low ethanol streams in integrated fermentation and separation processes (Cen 1993).

#### Energy and Costs:

Membrane based operations, as seen in Table 4.2, such as vapor permeation and pervaporation have the lowest amount of energy consumption per cubic meter of anhydrous ethanol produced (Li et al. 1992). Molecular sieve based ethanol dewatering operation consumes lower energy than conventional methods (azeotropic distillation, extraction etc) and costs less. This is essentially due to the reduction in operational costs. Membrane based operational costs are about 2 to 2.5 times lower than that for molecular sieve adorsption (Kaminski et al. 2008) and this is primarily due to the elimination of steam required for sieve regeneration. However, when dehydrated steam is recycled for regeneration operation large amounts of energy is saved and in these cases the energy advantage of membrane based technologies is reduced to only 4% (Leland 2008). Among various membrane separation techniques available, vapor permeation is apt for hybrid distillation-vapor permeation operations. It can be combined with distillation to produce fuel grade ethanol. Vapors rich in ethanol can be purified by attaching a modular unit to the distillation product line (Leland 2008). Since membranes process only high concentrations of ethanol, membranes foul less (Li et al. 1992). A liquid product, containing 99 vol% ethanol (Li et al. 1992) can then be obtained easily.

Even though membranes offer lower operational costs, the initial investments associated with membranes are much higher than those for molecular sieves. This is

primarily due to the high membrane prices associated with the module. Hybrid distillation-pervaporation profitability, especially for small scale processes, is not foreseen without reduced membrane costs (Kaminski et al. 2006). Even today molecular sieve adsorption remains the predominant technology used to dewater ethanol with vapor permeation and permeation becoming popular only very slowly.

The on-farm demonstration facility can be envisioned with <u>distillation to produce</u> <u>azeotropic ethanol and molecular sieve technology for the final dewatering step to</u> <u>produce fuel grade ethanol at >99 wt% ethanol</u>. Based on the technical study of various dewatering technologies the use of molecular sieves gives the farmer the opportunity to produce fuel grade ethanol at the most economical option available. Experiments conducted at the demonstration facility will be able to give answers regarding economics and energy efficiencies of the distillation step as well as final dewatering step. The first stage in the process of building the facility is establishing a unit capable of producing azeotropic ethanol. The design steps of this first stage are the focus of this thesis. Subsequent stages in the design will look at final dewatering.

# CHAPTER V

# ETHANOL DEWATERING: DESIGN BASIS

As seen, early work done for continuous on-farm ethanol production suffered several processing setbacks. The use of corn feedstock meant that the process consumed high quantities of water and energy. Several technical glitches were found in all on-farm ethanol operations including mechanical and structural failure of equipment, loss of ethanol, maintenance issues and start up issues. Knowing there can be several obstacles in the process of ethanol manufacturing on-farm one has to be careful during the design stage itself in trying to minimize any of these issues. The major driving forces behind design of the demonstration facility are:

# a) Obtaining the highest value product, anhydrous ethanol

This will ensure benefits from the sale of the product stay with farmers. The product should be able to meet the most stringent specifications for transportation fuel ethanol. However, in this thesis the design of the demonstration unit will focus on obtaining 95 vol% (or 190 proof) ethanol. The final dewatering step will be considered in subsequent investigations

# b) Mechanical simplicity and ease of maintenance

The selection of two column distillation for ethanol-water separation ensures that a simple scheme is being used. Fouling issues are contained in the beer column/stripper, continuous processing can be achieved and the process can be fine tuned to be energy efficient and reliable.

c) <u>Safety of the process</u>

This is also a major consideration during the design. This can be achieved by having an appropriate level of process automation for control and monitoring, relief systems and accessibility to emergency measures.

One advantage of using sweet sorghum is the processing simplicity found in obtaining the fermented juice containing 6-10 vol% ethanol. The yields of this fermented juice and the fermentation efficiency determine the amount of ethanol available and the total feed rate into the distillation step. For a 500 acre farm of sweet sorghum crop with yields of close to 30 ton/acre, 55% juice content, 15% sugar in juice, conversion efficiency in fermentation assumed to be 85%, the fermented juice calculated is close to 2,000,000 gallons (Holcomb 2008). A feed rate of 250 gal/h for a year round operation is appropriate for a small scale plant in order to process the amount of juice mentioned.

Keeping in line with other important issues such as conservation, an important feature of the proposed design is the emphasis on energy and water savings. The beer column design includes a forced circulation vaporizer. This design step will eliminate the need for fresh well water (for stripping steam) and reduce the total stillage produced. The vaporizer has been designed to utilize hot combustion gases from a natural gas fired combustor. Excess gases from this combuster can be utilized in a feed pre-heater. Along with this feature, hot stillage will also be used to heat the feed fluid to the required temperature. An alternative design option provides for the use of traditional stripping steam to concentrate ethanol in the beer column. Once the pilot facility is built the water

and energy savings can be quantified and compared. Other features to be incorporated into the separation step which result from major modifications of the 1980s design are

- To facilitate the use of non-traditional utilities a reboiler instead of stripping steam is used to produce the vapor stream. The reboiler has been designed as a shell and tube heat exchanger with hot combustion gases on the shell side of the heat exchanger. The gases are the product of combustion of natural gas in excess air.
- 2. Also, the condenser for ethanol product recovery has been designed as an air-cooled heat exchanger. This eliminates the use of cooling water and uses air as the cooling medium. The cooling medium is freely available and the added expenses of producing and treating the cooling water are eliminated.
- 3. The other altered attribute of our design is the rectifier and its design introduces several new features to the small scale concept. The use of trays or random packing commonly seen in the past has in this design been replaced by structured packing. Four trays at the bottom of the column, below the packing, provide additional functional benefits: a) these trays are intended to inhibit possible foulants from reaching the packing and b) they will facilitate removal of fusel components through a side draw. Fusels or fusel oils are a collection of higher alcohols which are found as fermentation products in small quantities (Lea et al. 1995). The side draw in the rectifier prevents the accumulation of these alcohols in the column which otherwise cause the separation performance to decline. Not seen in

early small scale demonstrations these could be the reason behind poor column performance.

To make the design process a reality entails two steps, the preliminary design and consequently the detailed design. The preliminary design stage includes putting together simulations, material and energy balances, equipment sizes and approximate costs for the process. Once the preliminary design is in place and the most economic option has been decided upon, the detailed design process begins.

The on-farm ethanol dewatering process consists of five major unit operations. This includes two distillation columns and associated heat exchangers and feed preheaters. There are twelve major streams in the process and six components considered including ethanol, water, acetic acid, lactic acid and glycerol. The design of the on-farm ethanol dewatering process was designed to meet the following conditions and assumptions:

- The plant will be designed to process 250 gal/h of feed. The design process was based on the processing of feed from sweet sorghum feedstock but the operation is not limited by the type of feedstock used.
- The feed has been assumed to contain 6.5 vol% ethanol. The analysis of feed performed by the department of Biosystem and Agricultural Engineering found that the feed contains a range of ethanol from 6.5 to 10 vol% ethanol. A conservative value has been taken for the simulation and subsequent design.
- 3. The final product has 94.2 wt% or 95.3 vol% or 190 proof ethanol.
- 4. The content of ethanol in the stillage is assumed to be 500 ppm.

- The processing system is recovery is at least 90% ethanol from the feed.
  The product rate is 15 gal/h.
- 6. The design includes a fusel draw from the rectifier with a nominal flowrate.
- 7. The rectifier is assumed to operate at atmospheric pressure.

# CHAPTER VI

# ETHANOL DEWATERING: DETAILED DESIGN

**VI.1 Simulations:** 

Simulations for the separation configurations were set up with Chemstations' CHEMCAD 6.1 and reflect a typical 'on-farm' or small-scale operation. A typical small scale arrangement usually consists of two columns, as shown in Fig 6.1. The first column, known as the "beer still" or "beer column" concentrates the ethanol from 6.5 to approximately 40 vol %. Product ethanol, (95 vol %) is obtained with the second column, also called the "rectifier."



Fig 6.1:Simulation arrangement (a) Traditional design employing stripping steam in the beer column (b) Alternative OSU design using a beer column reboiler and air-cooled condenser

The bottoms product from the beer column consists of mostly water, and is reused in the feed pre-heater to minimize wastage of energy. Bottoms from corn fuel ethanol processing plantare known as "stillage" and those from sugar based operations are called "vinasse." The bottom products from distillation of sweet sorghum fermented juice are expected to be closer to the composition of vinasse. However, in this report the two terms will be used interchangeably. In sugar based operations the vinasse produced has a solid content of 2-10% concentrated from the fermented juice feed into the bottoms (Cortez et al. 1997).

For simulation purposes a single tower can be considered for the process flow layout and is often done for simulation convergence ease. The beer column and the rectifying section represent the stripping (below feed point) and rectifying section (above feed point) respectively of a single column. The use of a single tower arrangement in real life is impractical, due to several reasons. One of the primary reasons is the size of a single column. As discussed previously, there are typically two columns each close to 30 feet tall. Combining the two could result in towers as high as 70 feet tall. For a farm scale operation this size makes it impractical in terms of construction and maintenance. The presence of large quantities of solids in the feed also makes the idea of two separate columns more appealing. High temperatures lead to protein deposits on column surfaces and internals (SERI 1982). In a two column arrangement, the fouling problems and corrosion of the tower are restricted to the stripping or beer column and thus this configuration has been widely adopted.

The third reason is that the use of trays and/or packing in the second column is possible. Arrangements where trays and packing have been used in a single column are

not uncommon industry practice. A combination of the two is often used to optimize separation achieved (Sulzer 2009). Fouling in the first column requires the use of trays, which are better suited for unclean applications. Packing can be used in combination with trays in the rectifier column. The use of a combination provides other advantages. Structured packing provides higher efficiencies and provides more capacity per unit volume than its tray counterpart. However, the advantage comes at a significant increase in cost per unit volume of packing (Seader et al. 2006). With the use of a combination, cost and efficiencies can be optimized. Thus from the point of view of an engineer as well as from somebody working on-farm, the two column layout seems more feasible and has been examined in detail.

The simulation of the on-farm separation scheme was set up with a fixed feed rate of 250 gal/h which is typical of small scale units (Nellis et al. 2000). The flowrate used in the design is limited by the design of the distillation columns. Both the columns are restricted to the diameter of 1 ft, the smallest diameter appropriate for a small scale set up. Operating at 75% approach to flood operation, results in a feed flowrate of 250 gal/h, for which the liquid and vapor flowrates in the column are within the hydraulic limit. Also, as discussed earlier based on a processing scenario where 2,000,000 gal (based on a 4000 gal/acre juice for a 500 acre farm) of fermented juice is produced a total of 333 days are needed to process that quantity of juice with a 250 gal/h flowrate.

The rectifier is simulated to operate at a little above ambient pressure. A pressure drop of 0.1 psia per stage is taken into account for the design. In the 1980s scenario design steam is introduced at the bottom of the beer column for concentrating ethanol as shown in Fig 6.1 (a). A total condenser is present with the rectifier to condense ethanol

product vapor to ambient conditions. Improvements discussed in the design have been captured in the alternate simulation which includes a reboiler which uses combustion gases for the heat supply, air cooled heat exchanger for the rectifier, fusel draws and inclusion of structured packing. This is shown in Fig 6.1 (b).

# VI.1.1 Thermodynamic Model:

The Non-Random-Two-Liquids (NRTL) model was used to estimate the thermodynamic K-values for the ethanol water system in the simulation and the Latent heat model was used to estimate enthalpies for the system. The components used in the simulation are shown in Table 6.1. The standard vapor reference state was at 60 °F. Atmospheric pressure in the software is 14.6959 psi.

Components	Chemcad ID #	Name	Formula
1	134	Ethanol	$C_2H_6O$
2	62	Water	$H_2O$
3	680	Lactic Acid	$C_3H_6O_3$
4	268	Glycerol	$C_3H_8O_3$
5	130	Acetic Acid	$C_2H_4O_2$
6	277	Succinic Acid	$C_4H_6O_4$

Table 6.1: Components used in simulation of on farm ethanol dewatering

The NRTL parameters are calculated according to the following equation

$$T_{ij} = A_{ij} + \frac{B_{ij}}{T} + C_{ij} \times \ln(T) + D_{ij} \times T \qquad \dots \text{Eq 6.1}$$

The interaction parameters by the software are shown in Table 6.2.

i	j	$\mathbf{B}_{ij}$	$B_{ji}$	α	$A_{ij}$	A <sub>ji</sub>	C <sub>ij</sub>	C <sub>ji</sub>	D <sub>ij</sub>	$\mathbf{D}_{ji}$
1	2	-55.17	670.44	0.303	0	0	0	0	0	0
1	4	398.44	79.51	0.296	0	0	0	0	0	0
1	5	-147.79	105.31	0.299	0	0	0	0	0	0
2	4	258.11	-274.35	1.011	0	0	0	0	0	0
2	5	424.02	-110.57	0.3	0	0	0	0	0	0

Table 6.2: Binary interaction parameters used by Chemcad

VI.1.2 Feed Composition:

The feed composition, representative of a typical sweet sorghum fermented juice sample, is shown in Table 6.3. Apart from these components, the feed has some solid content as well. The design of the industrial beer still accounts for the presence of solids and special tray designs such as the SVG trays which provide high fouling resistance (Sulzer 2009) will be employed. For our analysis, the solid content is taken into consideration only during the selection of column ancillaries and is not incorporated in the simulation.

Component	Composition	Typical ranges		
Component	vol %	g/L		
Ethanol	6.50	50 - 80		
Water	92.51			
Lactic acid	0.24	0-6		
Glycerol	0.35	3-6		
Acetic acid	1.86	0-4		
Succinic acid	0.02	0-0.6		

Table 6.3: Representative feed composition

### VI.1.3 Simulation Results:

For simulation of the traditional design using stripping steam in the beer column, the ethanol product concentration was 95 vol%. The number of trays on each column, and the steam rate was varied for the product and ethanol flow-rate in the stillage. The results are shown in Table 6.4, row three. The optimum number of stages was found to be 26, each column having 13 stages. The design pressure in the condenser was kept constant at atmospheric. The column and ancillary equipment were designed based on the data in table 6.4.

Design	Number of stages		EtOH in stillage	Steam	EtOH product stream	Condenser Duty	Feed HEX duty
Traditional Design: Stripping steam in	Column 1	Column 2	Gal/h	lb/h	Gal/h	MMBtu/h	MMBtu/h
beer column	13	13	1.40	558.3	15.58	-0.3792	0.1808
Design	Number of stages		EtOH in stillage	Reboiler duty	EtOH product stream	Condenser Duty	Feed HEX duty
Alternative OSU design: Beer column reboiler and air-cooeld condenser	Column 1	Column 2	Gal/h	MMBtu/h	Gal/h	MMBtu/h	MMBtu/h
	13	13	1.47	0.5973	15.58	-0.3630	0.1197

Table 6.4: Steady state output from simulation

The alternative arrangement, which incorporates a reboiler with the beer column, was simulated using CHEMCAD 6.1 as well. The optimum for this arrangement was with 13 theoretical stages (12 stages + reboiler) in the beer column and the same number for the rectifier. The steady state reboiler duty was 0.5973 MMBtu/h. The energy requirements of the system were investigated with the help of the simulations. Detailed CHEMCAD outputs of both simulations can be found in Appendix A1 and A2.

As discussed earlier the alternative OSU design incorporates the use of a reboiler with the beer column to avoid the use of stripping steam. The idea behind this is to minimize the use of well water and treatment chemicals needed for the operation of the boiler to produce the steam. Also with the introduction of the reboiler, the amount of wastewater generated will be limited to that in the fermented juice. As seen in Fig 6.2 (a) a preliminary estimate based on the simulation suggests a decrease in wastes by 12% when the ethanol concentration is 6.5 vol% in the feed.



Fig 6.2: Reduction in Waste: (a) % Decrease in liquid: Stripping steam vs. reboiler design (b) Stillage produced in both designs

Traditional reboilers like the kettle type or thermo-siphon reboiler use steam as a heating agent. The use of combustion gases to vaporize bottoms product provides a cleaner solution that does not introduce the need for the addition of water to the overall process.

The process flow diagram for the stripping steam case is presented in Fig 6.3. The process diagram for the reboilers case is presented in Fig 6.4.

# VI.2 Equipment Sizing

Preliminary equipment sizing includes the determination of the number of trays, finding the approximate sizes of the columns and the area required for the heat exchangers. The number of trays was calculated from the efficiency of the trays and the calculated number of theoretical stages shown in Eq 6.2, where  $E_0$  is the efficiency of the trays. The overall stage efficiency was provided by Sulzer Chemtech. A value of 56% was used for the beer column trays.

$$N_{trays} = \frac{N_{theoretical stages in column}}{E_o} \qquad \dots Eq \ 6.2$$

The height of the beer column tray section was calculated to be 29 ft. The diameter of the column was determined by the maximum approach to flood approach described in Seader and Henley (Seader et al. 2006). A value of 75% approach to flood was used and the column diameter was calculated to be 1 ft.

The heat exchanger area required was calculated from methodology described in the Handbook of Energy Systems Engineering (Bell 1985). The preheater, E-1, uses hot stillage to heat the feed to an intermediate temperature of 150  $^{\circ}$ F. The area required for this preheater was calculated to be 12.6 ft<sup>2</sup> for an incoming stillage temperature of 225  $^{\circ}$ F.





#### VI.3 Detailed Design:

VI.3.1 Beer Column and Rectifier:

The proposed farm-scale dewatering facility is intended to serve as a demonstration and a research facility. Sulzer Chemtech USA (www.sulzerchemtech.com), arguably the world's leading supplier of distillation equipment for bioethanol separation, has collaborated with us. Dan Summers, Sulzer's Manager of Tray Technology and bioethanol separations expert, has provided the detailed design for the two distillation columns at the heart of the dewatering system.

The beer column will utilize 23 Sulzer SVG trays (based on 56% tray efficiency). The trays will be 12 inch standard cartridge type (Sulzer standard Index #1) with a Teflon seal gasket. Tray spacing will be 15 inches to handle the desired loads. Total height of the beer column will be approximately 38 ft.

The rectifier column will use Sulzer M752Y structured packing for the bulk of the theoretical stages. The HETP for this packing is 14 inches in this service. The resulting 11 stages will need a bed that is approximately 14 ft deep. The bottom of the rectifier will have four cartridge trays to both protect the packed bed from solids carry-over from the feed and to enable two fusel draws. The tray efficiency of these four trays will be 50% (two theoretical stages). Total height of the rectifier column will be approximately 29 ft. Both columns will be constructed from standard 12 inch Schedule 20, 316 stainless steel pipes. The beer column will also be designed with a traditional stripping steam connection to provide multiple operating modes. Additional details for the design are given in Appendix B.

### VI.3.2 Heat Exchangers:

Both the vaporizer and rectifier column condenser have been designed by Prof. Ken Bell, a collaborator and world-recognized expert in the area of heat transfer. The condenser was designed as an air cooled heat exchanger. This was done to eliminate the use of cooling water. The ambient temperature for the design was assumed to be 100 °F. The resulting design consists of a tube bundle with 3 rows (16-17-16 arrangement). A 30 inch diameter fan with a ½ or ¾ HP motor provide the required air flow. The overall air-side pressure drop is 0.5 inch of water, which is a common design figure (Wilbur 1985). The face area of the heat exchanger is 3.1 ft x 4 ft. A detailed description of the calculations has been given in Appendix C.

The reboiler design is driven by the need for frequent tube side cleaning. In the reboilers, the bottoms product circulates through the shell and tube reboiler at very high velocities to keep solids present in the stream suspended. The reboiler was designed for only 1% vaporization per pass. For the production of combustion gases, natural gas will be used as a fuel source, which is readily available. A packaged air heater will be used to produce hot combustion gases at 600 °F. The hot gases will be present on the shell side of the heat exchanger with the liquid in the tubes. The tube bundle, consisting of 39 tubes each 6 ft long and 16 fins/inch, will provide the required heat transfer area. An 8 inch standard pipe will form the shell for the heat exchanger and the tubes will have an outside diameter of ¾ inches. Calculations have been detailed in Appendix C1. The heat exchanger has been designed for 1% vaporization of fluid taking place inside the tube with most of it occurring in the line leading to the column in order to minimize fouling of the tubes. The deposition of the solids is then concentrated in the line, making it more accessible and easier to clean.

VI.3.3 Packaged Air Heater:

The packaged air heater considered is manufactured by Stelter and Brinck and includes a duct section, skid, process blower, inlet and outlet transitions, gas valve train, safety controls, temperature controls and high temperature limits (Brinck 2009). The unit is pre-piped, pre-wired and, ready to fire when it arrives on site. The equipment is restricted to operational pressure limit of 14.69 psig. Process air temperatures of 1200 °F can be generated in the output air streams with low NO<sub>x</sub> emissions. As an alternative to using packaged air heater, direct fired burners can be used to produce a stream of hot gases from burning natural gas. Maxon provides low temperature burners capable of producing gases up to 1000 °F (Maxon 2009).

### VI.3.4 Instrumentation:

The control strategy for the OSU Bioethanol Pilot Plant is presented in Fig 6.6. Control valves, pressure, level and temperature transmitters and indicators for the process have been quoted by Rosemount Inc. Mr. Warren Meyer, Senior Sales Representative at Emerson Process Management/Rosemount Inc. has provided a list of appropriate instrumentation based on the requirement of the project and is shown in Table 6.5. These include pressure, temperature, level and flow transmitters. Orifice plates and isolation valves needed for the pressure transmitters have also been included. Control valves, listed in Table 6.6 for the process, have also been quoted by Emerson Process Management. Final recommendations will be made by Mr. Paul Behling, Inside Sales Specialist from Vinson Process Control.



Fig 6.5: Piping and Instrumentation Diagram: OSU Bioethanol Facility
Tag	Туре	Rosemount Model No.	Remark
PT-2,9,100,121,122,129	Pressure Transmitter	2051	Max pressure range: 0 -800 psi, Min pressure range: 0-8 psi. Inbuilt pressure indicator
	Manifold Isolation valve	306	
FE-1, 3, 4, 5, 121, 122, 128, 63, 203, 64, RO-129	Integral orifice flow element	1195	Contains both piping and orifice plate
FT-1, 3, 4, 5, 121, 122, 128, 63, 203, 64	Pressure flow transmitter	2051C	Inbuilt flow indicator
TT-101, 103, 65, 67, 110, 7, 9, 8, 63, 101, 22, 39, 126, 128, 4, 43	Temperature transmitter	644	Inbuilt temperature indicator
LT-201, 202, 203, 1, 2, 4	Level transmitter	3301	Inbuilt level indicator

 Table 6.5: Instrumentation Details for OSU Bioethanol Pilot plant

Baumann 24,000 Valves with PTFE plugs can be use for the 1 <sup>1</sup>/<sub>4</sub> inch liquid lines.

Recommendations are yet to be made for the gas and steam lines.

Count	Tag	Service	Fail
1	LCV-1	T-1 bottoms level	Closed
2	LCV-2	T-2 bottoms level	Open
3	LCV-4	D-4 (accumulator) level	Closed
4	FCV-4	T-2 reflux flow control	Open
5	FCV-1	Stripping steam to T-1	Closed
6	FCV-121	Natural gas feed to combustor	Closed
7	FCV-122	Combustion air feed to combustor	Open
8	TCV-128	Hot gas temp control	Open
9	FCV-3	Hot gas to E-3 (reboiler)	Closed

Table 6.6: Control Valves required for the Bioethanol process

# VI.4 System Hydraulic Analysis:

The hydraulic analysis of any process includes the selection of appropriate piping and pumps as well as determining the hydraulic capacity of the process. The idea behind the hydraulic analysis is to find out if there is enough driving force for fluid flow. This analysis also finds whether a chosen pump in the process will deliver fluids at required flow rates, with the selected lines and equipment.

At steady state the available driving force for flow is equal to the required driving force. The required driving force is calculated from process conditions and contains static and dynamic portions. The static portion does not vary with fluid flow rate and includes the potential head difference and the pressure differential between the source and the destination of the pipe. The dynamic portion of the driving force includes the pressure drop across the open control valve and orifice as well as line losses, all of which vary with the velocity of the fluid in the line.

The system curve is then compared against the  $\Delta P_{available}$  from pump data to establish the hydraulic capacity of the system, i.e. the delivery flow rate in the process section with the selected pump.

### VI.4.1 Line Sizes and Loss Calculations:

The size of piping selected is based on pressure drop or calculated fluid velocity in the line. Typical velocities for a thin liquid in a process line lies between 4 and 8 ft/s (McCabe et al. 2001). For smaller processes, such as the OSU Bioethanol pilot plant, lower velocities and low line losses can be expected. The selection of the pipe diameter in the process is also limited by the minimum viable diameter. Lines carrying feed or stillage are restricted by the presence of solids in the stream. A minimum pipe size of 1 <sup>1</sup>/<sub>4</sub> inch is preferred for such streams in the process. Smaller diameter pipes are more likely to plug, provide less rigidity and were not considered for feed lines. Lines with ethanol product and cleaner fluids can use smaller diameter pipes and the 1 inch diameter pipes were selected for these.

The flow of fluids in pipeline and through fittings is associated with friction losses. Friction losses are a result of the following (Crane 1986):

- 1. Pipe friction: Due to the rough surface of pipes
- 2. Directional changes in flow: Pipe bends, tees, bends and elbows
- 3. Flow obstructions: Orifices and valves
- 4. Cross sectional area changes: Pipe entrances and exits, sudden enlargements and contractions

Changes in velocity occur due to the presence of these obstructions in the length of the line. The loss in pressure due to the collective effect of these factors is captured in head loss or  $h_L$  in Eq. 6.3 where K is the total resistance coefficient.

The value of K for fittings calculated from equations described in Crane Technical Paper No. 410 (Crane 1986). The friction losses in the pipeline were calculated for each individual pipe section based on the number and type of fittings and the length of the pipe. This was done by creating Excel spreadsheets for each section. Each spreadsheet has input fields for fittings, length of pipe, fluid and pipeline details. The velocity of the fluid and the hydraulic friction losses are calculated and displayed as results. The results have been presented in Table 6.7 below.

Line No.	From	То	Temp	Total Flowrate	Fluid sp. gravity	Density	Viscosity	Volm flowrate	Schedule 40Pipe Size	Vel	ΔP
			_		(w.r.t	2					
			°F	lb/h	water)	lb/ft <sup>3</sup>	cP	gpm	inches	ft/s	psi
1	Tk-1	P-1	60	2,062	0.988	61.62	1.162	4.2	1 1/4	0.881	0.2407
2	P-1	E-1	60	2,062	0.988	61.62	1.157	4.2	1 1/4	0.895	0.2844
3	E-1	T-1	120	2,062	0.976	60.85	0.583	4.2	1 1/4	0.907	0.0622
4	E-4	D-4	173	911	0.747	46.62	0.426	2.4	1	0.906	0.0307
5	D-4	P-4	120	911	0.776	48.42	0.681	2.3	1	0.867	0.0168
6	P-4	T-2	120	806	0.776	48.42	0.681	2.1	1	0.769	0.1561
7	P-4	D-3	120	105	0.776	48.42	0.681	0.3	1	0.100	0.0015
8	T-2	P-3	186	587	0.805	50.19	0.354	1.5	1 1/4	0.313	0.0017
9	P-3	T-1	188	587	0.805	50.15	0.351	1.5	1 1/4	0.313	0.0273
10	T-1	P-2	218	2,516	0.953	59.44	0.260	5.3	1 1/4	1.132	0.2104
12	P-2	E-1	225	2,516	0.953	59.44	0.260	5.3	1 1/4	1.132	0.0761
13	E-1	D-3	154	2,516	0.980	61.11	0.428	5.1	1 1/4	1.101	4.2082

Table 6.7: Line sizing: Calculated line losses and velocities (incompressible fluids)

Lines carrying compressible fluids were modeled as multiple short segments of incompressible flow and the non-recoverable pressure drop was calculated by numerical integration over the length of the pipe. The results are given in Table 6.8.

Line No.	From	То	Total Flowrate	Specific volume	Viscosity	Mass flowrate	Schedule 40Pipe Size	Vel	ΔΡ
				3					
			lb/h	ft³/lb	cP	lb/ft² s	inches	ft/s	psi
15	T-1	T-2	704	12.99	0.0115	3.8	3	49.48	0.114
16	T-2	E-4	953	10.30	0.0106	5.2	3	53.13	0.109
17	B-1	T-1	558	3.09	0.0156	14.9	1 1/4	46.09	0.877
18	Natural Gas	C-1	38	7.46	0.0115	1.0	1 1/4	7.58	0.012
19	Air	C-1	4,687	1.68	0.0191	25.4	3	42.63	0.630
20	C-1	E-3	4,725	11.49	0.0331	25.5	3	294.16	4.865

Table 6.8: Line Sizing: Calculated velocity and pressure drop (compressible flow)

The accuracy of spreadsheet calculations was checked against pressure drop values for water and air (incompressible and compressible) through 100 ft of pipe obtained from Appendix B-14 and 15 of the Crane Manual. Results for the flow of 4 gpm water at 60°F through a schedule 40, 1 ¼ inch pipe were obtained within a percentage

error of 2%. For compressible flow when compared against the flow of 2.563  $ft^3$ /min air in similar conditions the results were within 9% error.

#### V.4.2 Pressure Drop across Control Valve and Orifice:

For the purpose of hydraulic capacity analysis the position of the valve is given by the function f (l) assumed to be 1 for a fully open valve. The pressure drop across the valve can then be calculated by Equation 6.4 (Seborg et al. 2004)

$$\Delta P_{valve} = \left[\frac{q}{C_v \times f(l)}\right]^2 \times sp. g \qquad \dots \text{Eq. 6.4}$$

In the above equation,  $C_{\nu}$  is the maximum value of the valve coefficient for a valve and defines its capacity.

Pressure drop or differential pressure across the orifice depends on the model selected. The Rosemount integral orifice plates can be ordered for a differential pressure range of 0 to 1 psi. For our analysis, we will assume the permanent pressure loss across the orifice to be 1 psi at the design flow rate.

## V.4.3 Hydraulic capacity:

The pump selected for the feed system is a MARK centrifugal 82M3 low flow ANSI centrifugal pump. The NPSH<sub>required</sub> for this pump is 2 ft. The impeller size used in the analysis is 8.19 inch. The electric motor rating of the pump is 1 HP. The hydraulic analysis shows that the flow rate expected when the valve is fully open is 10 gpm, higher than the design steady state flow as shown in Fig 6.6 displayed below. The required steady state flow rate of 4 gpm can be achieved when the control valve is throttled to 35% open position.



Fig 6.6: Hydraulic Analysis of the feed section of the process

## VI.5 Costing

Economic estimates were made for the beer column, rectifier and associated auxiliary equipment. The two cases a) traditional design employing stripping steam b) alternative OSU design using beer column reboiler and air-cooled condenser have been compared for equipment costs for the year 2009.

These cost estimations were done based on budgetary quotes from suppliers and manufacturers. Internet pricing was used wherever a direct quote could not be procured. The total purchased cost for all equipment for the two cases are tabulated in Table 6.9

Table 6.9: Comparison in Equipment Purchased Cost						
	Traditional Stripping	OSU Alternative				
Equipment	Steam Case	Reboiler Case				
Instrumentation: Control	57,500	76,500				
Instrumentation: Measurement	2,900	4,200				
Column body and Internals	49,500	49,500				
Boiler	16,000	-				
Combuster	-	38,000				
Heat Exchangers	2,700	6,200				
Pumps	3,700	3,700				
Storage Tanks	36,500	36,500				
Total	168,800	214,600				

For the traditional design scheme using stripping steam the total heat exchanger cost includes the cost of the boiler in addition to the feed pre-heater and condenser. The OSU design alternative reboiler case includes the packaged air heater cost and the reboiler cost. The purchased equipment cost includes storage tank costs for both cases. The cost of the storage tanks and instrumentation are a large percentage of the total costs with instrumentation at 35% and tanks at 17 -21 % of the total. The OSU alternative design with the reboiler is more expensive than the stripping steam layout. Details for each equipment selection are given in Table 6.10 to Table 6.15.

<b>T</b>	<b>N T</b>	-		<b>TT 1</b> .	Price	Total
Equipment	Name	Tag	Remarks	Unit	(\$)	Price (\$)
			Includes: 12" flanges x 3, 2"			
			feed nozzle x 1, 6" overhead			
			nozzle x 1, 5' top section, wire			
			wound gasket for upper			
			section flanges, bolting. 12"			
	Beer		vessels, 304 L Schedule 20			
Column Shells	Still	T-1	pipe, 1480 lbs	1	14,300	14,300
			Includes: 12" flanges x 3, 2"			
			nozzles x 3, 4" nozzle x 1 at			
			top, 5' top section approx, wire			
			wound gasket for upper			
			section flanges, bolting. 12"			
			vessel, 304 L Schedule 20			
	Rectifier	T-2	pipe, 1180 lbs	1	11,200	11,200
	Rectifier	T-2	Distributor	1	2,000	2,000
			Bed Support	1	1,000	1,000
Column						
Internals			M752Y Packing	1	2,500	2,500
			Cartridge trays	4	1,063	4,250
	Beer		· ·			
	Still	T-1	Cartridge trays	23	620	14,250
					Total	¢ 10 500
					rotar	φ 42,3UU

Table 6.10: Column Pricing Details Provided by Sulzer Chemtech

Unit

Company	Equipment	Tag	Details	Unit	Unit Cost (\$)	Total Cost (\$)
Federal Corporation	Boiler	B-1	HE15 Lattner vertical high pressure boiler. 150 psi. Natural gas burner including all operating and safety controls. 15 BHP. Quote includes feed water system (tank, water gauge, make-up valve, pump) and blowdown separator and after cooler.	1	15,636	15,636
Stelter Brinck Quotes	Combuster	C-1	Includes duct section, skid, process blower, inlet & outlet transitions, gas valve train, safety controls, temperature controls and high temperature limits. Unit is pre-piped and prewired, ready to fire on site arrival. Test fired before shipment	1	38,000	38,000
Cool-Line Internet Pricing	Rectifier Condenser Air Cooled HEX	E-4	55 HP Forced Air Oil Cooler, AC Motor, Flow Ranges 8 to 80 GPM, Max pressure 3771 psi, Max Temp 250 °C, Voltage 230/460, Motor Amps 6.2/3.1, 2, Aluminum, 26" x 30" x 20"	1	1,799	1,799
Love Joy Internet Pricing	Feed Preheater: Double pipe	E-1	Flow Ranges 10 to 87 GPM, HP Heat Removed 35 to 130, Max Oil Pressure 580 PSI, Max Water Pressure 230 PSI, Cooling Surface 82.6 ft <sup>2</sup> , aluminum fins, copper tubes, Rotating mounting brackets, Removable end cap, 8" x 30" x 7"	1	934	934
Louisiana Chemical Equipment Company Quote	Reboiler: U- Tube	E-3	Used, 82 sq ft area, Max pressure in Shell and Tube 150 psi, Max temp S & T 400 °F, 1" dia tubes, 1' 4" shell dia, Tube length 6' 6".	1	3,500	3,500

Table 6.11: Heat Exchanger Price and Details

Some equipment prices obtained are for used equipment. These prices are readily available and in some cases used equipment dealers were contacted for direct quotes on suitable material available from their inventory.

Company	Equipment	Tag	Remarks	Unit	Unit Price (\$)	Total Price (\$)
Dayton Inc. Internet Pricing	Pumps	P-1, P-2, P-3, P-4	Self Priming Centrifugal Pump, HP 3/4,Volts 115/230, Full Load Amps 10.8/5.4, Material (impeller, shaft, housing) 316 SS, Inlet/Outlet 1.5", Max Solids Dia 0.125 In, Temp Range 40 to 200 F, Seal Carbon Ceramic Viton, Maximum Pressure 165 PSI, Max Temp 200 F, 16 7/8 " x 9 1/8 "	4	914	3,656

# Table 6.12: Pump Costs and Details

It should be noted here that pump selection for all the four cases were done separately. In some cases however, pump selection software could not gives matches for required flow. Pump selection is being looked into with greater depth.

					Unit	
					Price	Total
Company	Equipment	Tag	Details	Unit	(\$)	Cost (\$)
			Philips Steel Fabricators Inc.			
			10,000 gallon non-jacketed			
Equipment			horizontal storage tank, 316 L,			
Internet	Feed and	Tk-1,	15 psi, 250 °F, Dished top and			
Pricing	Stillage	Tk-2	bottom, 8' dia, 26' TT	2	12,500	25,000
			1200 Gallon Vertical Sanitary			
Internet	Condensate		Stainless Steel Tank, 72" dia x			
asking	Storage		66" straight side, dished bottom			
price	Tank	D-4	head, center bottom outlet	1	4,500	4,500
			Used S/S 5000 Gallon Storage			
			Tank, Vertical, Flat Top, 304			
			S/S, Slight Dish Bottom, on (4)			
			XH H Beam Legs, 9' Dia x 11'			
Machinery			Straight, 13'6" OAH, Top			
and			Openings: 20" Top Manway,			
Technolog			(2) 2" Top Openings, (1) 2"			
y Group	Ethanol		CBO, (3) Support Rings, prior			
LLc.	product tank	D-3	use: latex paint	1	7,000	7,000
					Total	\$ 36,500

# Table 6.15: Storage Tank Costs and Details

			No.	Unit	
	Rosemount		of	Price	Instrument
Type of instruments	Model No.	Tag No.	units	(\$)	price (\$)
		PT-2; 9; 100;			
D	2051	121; 122;	6	0.017	10 100
Pressure transmitter	2051	129	6	2,017	12,102
		PT (IV) -2; 9;			
Manifold isolation value	306	100; 121; 122; 120	6	172	1 032
	300	$\frac{122, 129}{\text{EE}  1 \cdot 4}$	0	172	1,052
		$\Gamma E = 1, 4,$ 121, 62,			
Internal orifica element	1105	121,03;	5	2561	12 905
Integral ornice element	1195	203 EE (II) - 2:	5	2,301	12,805
		FE (U) - 3;			
0.5	1400	5; 122; 128;	~	410	0.065
Orifice flange union	1496	04 FF (D) 2	3	413	2,065
		FE (P) - 3;			
	1.405	5; 122; 128;	_		1 1 1 0
Orifice plate	1495	64	5	222	1,110
		FT - 3; 122;			
		128; 64; 1;			
		4; 5; 121;			
Pressure flow transmitter	2051C	63; 203	10	2,242	22,420
		FT (IV) - 3;			
		122; 128;			
		64; 1; 4; 5;			
		121; 63;			
Manifold isolation valve	305	204	10	548	5,480
		TT-101; 103;			
		65; 67; 110;			
		7; 9; 8; 63;			
		101; 22; 39;			
<b>T</b>	C 1 1	126; 128; 4;	16	1 000	17 400
Temperature transmitter	644	43	16	1,088	17,408
Lovel Transmitter	3301	L1-201; 202;	6	2 070	19 400
	5501	203; 1; 2; 4	U	3,070	10,420
		$201 \cdot 202 \cdot$			
Readout for level transmitter	751	203: 1: 2: 4	6	727	4.362
	. = -	, _, _, _, ,	~	Total	\$ 97 201
				Total	φ 21,404

Table 6.14: Instrumentation for Control: Quotes provided by Rosemount Inc.

					Unit	Total
	Ashcroft	Temperature			Cost	Cost
Instrument	Model No.	Range	Tag No.	Units	(\$)	(\$)
Thermometer						
and		200 - 1000	TI-129, TW-			
Thermowells	50EI60E040	°F	102, TW-64			
			TW-129, TW-			
	50W0250HT260S2		102, TW-64	3	125	374
	50EI60E025	50 - 400 °F	TI-111			
	50W0162ST260S2		TW-111	1	117	117
			TI - 10; 5; 41;			
			201; 202; 203;			
	50EI60E180	50 - 300 °F	8; 11; 125			
			TI - 10; 5; 41;			
			201; 202; 203;			
	50W1650ST260S2		8; 11; 125	9	204	1835
			TI-6; 62; 21; 23;			
	50EI60E025	50 - 300 °F	40; 41; 42; 38			
			TW-6; 62; 21;			
			23; 40; 41; 42;			
	50W0162ST260S2		38	8	117	933
	50EI60E025	$100$ - $800\ ^{\rm o}{\rm F}$	TI - 66; 102			
	50W0162HT260S2		TW - 66; 102	2	117	233
			PI - 1; 21; 22;			
			100A; 102; 62;			
			2A; 3; 121A;			
			122A; 123; 124;			
			129A; 101; 3R;			
Pressure			9; 10; 11; 8;			
indicators			203; 67; 64	22	57	1,254
					Total	\$ 4,746

Table 6.15: Instrumentation: Measurement

As discussed earlier, the cost of the instrumentation comes out to be a large percentage of the total cost. Also notably large is the cost of the stainless steel storage tanks. A limiting factor in the selection of any equipment factor is material compatibility. From interaction with ethanol industry insiders and design engineers it was found that the industry wide standard for all process equipment is 316 stainless steel. These need stems from the presence of water and trace acids in the process. These substances corrode carbon steel and thus equipment made from carbon steel is not preferred in the ethanol industry. Use

of stainless steel also makes the capital cost of the plant high and is reflected in the pricing.

#### VI.5.3 Break-Even Analysis

Our interests, in this analysis, are limited to just the ethanol dewatering section of the plant. Therefore, the harvesting, fermentation and waste treatment are not included in the analysis. A cash flow table was set up to understand the project economics and conduct the break-even analysis. The project life was assumed to be 5 years with a minimum rate of return of 15%. The ethanol concentration in the product stream was 95.3 vol% and the outflow of the product from the system was fixed at 15.58 gal/h.

The break-even analysis (base case) is for 333 days of operation, 24 hours a day. As discussed earlier, this value is based on a 500 acre farm producing 4000 gal/acre of juice (4000 gal/acre x 500 acre = 2,000,000 gal of juice). The number of days is based on a 250 gal/h fermented juice feed, for a 24 hour/day plant operation (2,000,000 gal juice/(250 gal/h x 24 h/day) = 333 days).

The equipment cost was based on information from direct quotes and represents realistic values. Installation costs were also factored into the fixed capital costs. For a typical chemical plant the installation costs vary between 25 to 55 % (Peters et al. 2003) (of purchased equipment cost) and a value of 49% was taken for the pilot plant. This includes consideration for labor, material and piping costs. Details for the fixed capital costs are given in Table 6.16.

Table 6.16: Fixed Capital Cost						
	Traditional Stripping	OSU Alternative				
Equipment	Steam Case	Reboiler Case				
Instrumentation: Control	57,500	76,500				
Instrumentation: Measurement	2,900	4,200				
Column body and Internals	49,500	49,500				
Boiler	16,000	-				
Combuster	-	38,000				
Heat Exchangers	2,700	6,200				
Pumps	3,700	3,700				
Storage Tanks	36,500	36,500				
Equipment costs	168,800	214,600				
Installation costs						
(@ 49% Equipment costs)	82,700	105,200				
Fixed capital costs	251,500	319,800				

The break even processing cost was calculated using the solver function in XL for a zero net present value (NPV). This represents the price per gallon of 190 proof ethanol which would be required to achieve a 15% after-tax rate of return (break-even point). The cash flow table (traditional design using stripping steam in beer column) is seen in Table 6.17.

The break-even price for the stripping steam design (total fixed capital investment of \$ 251,500) is \$ 1.01 per gallon of 190 proof ethanol. The break-even price for the reboilers design case (total fixed capital investment of \$319,800) is \$ 1.30 per gallon of 190 proof ethanol.

End of Year		1	2	3	4
	2009	2010	2011	2012	2013
Beer tower OH production, gal/hr	15.58	15.58	15.58	15.58	15.58
Beer tower OH production, kgal	118.7	118.7	118.7	118.7	118.7
Processing cost, \$/gal 190 proof	1.01	1.01	1.01	1.01	1.01
Processing revenues, k\$	120.0	120.0	120.0	120.0	120.0
–Op Costs	(24.9)	(51.3)	(52.8)	(54.4)	(56.0)
-Depreciation	(50.3)	(80.5)	(48.3)	(29.0)	(29.0)
-Writeoff					(14.5)
Taxable Income	44.8	(11.8)	18.9	36.6	20.5
-Tax @ 40%	(17.9)	4.7	(7.6)	(14.7)	(8.2)
Net Income	26.9	(7.1)	11.3	22.0	12.3
+Depreciation	50.3	80.5	48.3	29.0	29.0
+Writeoff					14.5
–Working Capital					
–Fixed Capital	(251.5)				
Cash Flow	(174.3)	73.4	59.6	51.0	55.8
Discount factor (P/Fi*,n)	1.0000	0.8696	0.7561	0.6575	0.5718
Discounted Cash Flow	(174.3)	63.9	45.1	33.5	31.9
Present Worth Cost @ i* =	0.00	k\$	(Solve for zero)		

Table 6.17: Cash flow table for OSU Bioethanol Pilot Plant: Steam Case

The operating costs include, maintenance and cleaning at 3% of fixed capital investment. For the traditional design using stripping steam in the beer column the operating costs included the following, as seen in Table 6.18.

					Annual
Direct Operating Costs - excluding					Cost
raw materials	Usage				\$/yr
Utilities	Utility use \$/unit			Steam	
Electricity (distillate, bottoms, reflux					
and feed pumps, boiler blower)	1.66	kW-hr	0.09	\$ / kW-hr	1,307
Boiler fuel cost	678	SCFH	7.88	\$/1000 SCFH	40,925
Boiler water cost	67	gph	2.99	\$ / kgal	1,531
Total Utility Costs					42,233
Maintenance and repairs (3% of fixed capital)					
Total Annual Operating Expenses (\$/yr)					

Table 6.18: Operating costs: Traditional design - stripping steam in beer column

Water treatment costs (water softening unit, chemicals, pumps etc) required for boiler feed water were not included in the fixed costs. However, the use of city water utility rates in the operating costs reflects the cost of water treatment in the analysis. Boiler water costs have been calculated based on water utility rates in Stillwater, Oklahoma (stillwater.org 2009). These rates include the costs for filtration, chemical treatment and disinfection. The electricity costs are also for the Stillwater area (general service, averaged for summer and winter month).

Operating costs for the OSU design case includes electricity (distillate, bottoms, reflux, feed pump motors, and motor for the air cooled heat exchanger fan), fuel costs for the combuster, and maintenance and repairs expense (also 3% of fixed capital).

Table 6.19: Operating Costs: OSO Design – Beer column reboner, air cooled condenser						
Annual						
Cost						
( <b>\$/yr</b> )						
Reboiler						
1,363						
54,277						
55,640						
<i>9,593</i>						
Total Annual Operating						
65,233						

Table 6.19: Operating Costs: OSU Design – Beer column reboiler, air cooled condenser

The total annual operating costs for the OSU design case with beer column reboiler and air-cooled condenser is \$ 65,233/year.

#### Sensitivity Analysis:

The base case economic analysis was based on the fermented juice output of a 500 acre farm. However, it is possible for farmers to have smaller farms. For a 160 acre farm producing sweet sorghum juice at 4000 gal/acre, the total juice produced is 640,000 gallons. The number of days the plant would need to be operational (24 hours/day), to process the juice, would be approximately 106 days. For a farm of 110 acres the plant would be operational for only 72 days. In each of these scenarios the economics of the on-farm unit will change.

If sweet sorghum juice yields are 2,000,000 gal/year (based on a 4000 gal/acre juice yield for a 500 acre farm), the plant can be and will have to be operated for more than 300 days to process the entire quantity of juice. Once the feedstock is harvested and fermented, the distillation can be carried out in a continuous fashion with interruptions only for cleaning and maintenance. Fig 6.8 shows the reduction in processing costs possible with extended operating period of the pilot plant.



Fig 6.8: Sensitivity Analysis - Break even processing cost vs. days of operation

For plant operation lasting 72 days, the processing costs are fairly high. As seen from the figure the processing costs can be lowered below \$ 1.5/gal 190 proof ethanol product with operation of the pilot plant or on-farm facility (using stripping steam or beer column reboilers and air cooled heat exchanger) for 365 days. The operation of a plant for all 365 days is unrealistic (as the plant will experience downtime, periods of shut-down for maintenance or cleaning activities). It can be thought of as the best case for the operation of the pilot plant.

The above cost estimates do not include the cost of growing, harvesting, and fermenting the sweet sorghum. Recent studies (Lemos 2009) have estimated the price of azeotropic ethanol between 2.75 - 2.95 \$/gal 190 proof ethanol. For the process to make economic sense the cost of the processing should be considerably lower than this estimated market price for 190 proof industrial ethanol.

The economic evaluation of on-farm ethanol dewatering in this study are based on realistic estimates. The break-even analysis and sensitivity study reveal the positive aspects of the project economics. In an optimistic scenario, where the on-farm ethanol distillation unit operates for 333 days a year, at 250 gal/h feed rate, the processing costs can be as low as \$ 1.01/gal 190 proof ethanol product. Experimentation on a demonstration facility will strengthen these conclusions and bring us closer to establishing the 'true' cost of on-farm ethanol dewatering.

## CHAPTER VII

#### SWEET SORGHUM FERMENTED JUICE ANALYSIS

To implement a successful design it is important to understand the characteristics of the sweet sorghum fermented juice or beer. Experiments were designed and conducted to get data for each attribute of the beer. The important characteristics of interest are:

- a) Solid content and fouling tendency
- b) Fusel alcohol content

## VII.1 Solid Content and Fouling:

Temperatures expected in the beer tower range between 173 °F and 212 °F. This increases the probability of protein and sugar deposition on the walls of the column and the trays. These organic residuals in the fermented juice, at temperatures as high as those seen in the beer column, "caramelize" and form a polymeric layer on exposed surfaces (Kenneth 2004). Along with temperature, the effect of pH has a significant role in the fouling tendencies of the beer. The lower the pH the higher is the deposition expected. Studies conducted on corn stillage show a higher protein deposition for lower pH levels of 3.5 than 4.0 or 4.5 (Wilkins et al. 2006).

Filtration experiments were conducted to determine the solid content of the beer. Well mixed beer samples were filtered through Whatman 42 filter paper. The difference in the weight of the dried filter paper was the solids contained in a fixed volume of the beer tested.

$$w_{solids} = w_{filter+solids} - w_{filter}$$
 .....Eq 7.1

$$\% \ solids = \frac{w_{solids}}{w_{solution}} \times 100 \qquad \dots \text{Eq 7.2}$$

Where,  $w_{solids}$  is the weight of the solids dried up on the filter paper.  $w_{filter+solids}$  is the weight of the filter paper and solids, dried.  $w_{filter}$  is the weight of just the filter paper.  $w_{solution}$  is the weight of the fixed volume of sweet sorghum fermented broth taken for analysis. Two different beer samples were tested and three draws were made from each sample. After filtration the filter papers with the solid samples were dried overnight to ensure the removal of any residual water.

#### VII.1.1 Solid content estimation:

The solids estimated in the sweet sorghum fermented juice were found to be low. A solid content of less than 2% was found in the samples tested. Detailed results are displayed in Table 7.1.

					U		
				$w_{solids}$ +	Dry Weight of		
Sample	$v_{liquid}$	$W_{liquid}$	W <sub>filter</sub>	$w_{\text{filter}}$	solid		Whatman filter
No.	(ml)	(g)	(g)	(g)	(g)	%Solids	paper
a		8.60	0.642	0.734	0.092	1.07	42
b	10	6.92	0.642	0.733	0.091	1.32	42
с	10	6.70	0.638	0.729	0.091	1.36	42
d	5	4.65	1.142	1.216	0.074	1.59	1
а	10	7.86	0.637	0.760	0.123	1.56	42
b	10	8.33	0.645	0.757	0.112	1.34	42
с	10	6.86	0.647	0.732	0.085	1.24	42
d	10	8.11	1.075	1.174	0.099	1.22	1

Table 7.1: Solid content in sweet sorghum beer tested

This is much lower than the water insoluble solid content found in corn which could be as high as 10% of the total volume (Öhgren et al. 2006). The smallest sized particles retained on the Whatman 42 filter were greater than the pore diameter of 2.5  $\mu$ m. The average solid content was determined to be 1.34 ±0.18 wt%.

The solids particles were very fine and formed a smooth but sticky layer on the surface of the filter paper. The filtrate obtained was a clear colored solution. The pictures in Fig 7.1 show the solid residue and the filtrate.



Fig 7.1: Solids determination experiment. (a) and (b) Solid residue on filter paper and (c) experimental filtrate.

The stickiness of the solid residue was thought to be coming from glucose and other sugar elements in the sweet sorghum broth.

# VII.1.2 Fouling Experiment:

For the fouling experiments, a stainless steel tube insert, shown in fig 7.2 (b) was chosen to mimic the surface of the column and ancillaries. The beer was heated in a beaker placed on a heating mantle and once it reached a constant temperature, the mimic surface was immersed into the liquid.



Fig 7.2: (a) Fouling experimental set up (b) Stainless steel tube insert (c) Top view of inset

Temperature was monitored continuously and additional beer was added to the beaker every 10 min to maintain nearly constant liquid volume and solid content.

The deposition of the solids was calculated on an area basis. pH was measured initially (pH = 4.0) and at the end of experimentation. The experimental set up is shown in Fig 7.2 (a) and Fig 7.2 (b) and (c) show the tube insert used.

The sweet sorghum fermented broth boiled at a constant temperature of  $91^{\circ}$ C. An increase in weight of the tube insert was observed. The weight of the tube insert was found to increase by 0.0385 % on an average. Fig 7.3 (a) shows the tube insert after the experiment. The brown spots on the rim are deposits from the sweet sorghum juice.



Fig 7.3: Fouling Experiment: (a) Tube insert, solid deposition on top rim clearly seen (b) Broth after experiment, scum like material on surface (c) Deposits on the thermometer

The experiment was performed twice and both times deposition of a layer of solids was on the surface of the inserted thermometer, as seen in Fig 7.3 (b). This was again, sticky to touch and required external effort with some kind of brush to remove. On a per unit area basis, the increase in solid deposition was  $3.585 \text{ g/m}^2$ . To evaluate the severity of this deposition a comparison was made with experimental results from other fouling studies. There are several differences in the fouling medium, experimental set up, and method of these experiments, however the information from these literature examples give an interesting perspective on the scale of the fouling issue.

This amount of deposition experienced in the above experiments was similar to that seen on 316 SS surface in experiments conducted by Belmar-Beiny et al (Belmar-Beiny et al. 1993). In these flow experiments (Re =5200) lasting one hour, conducted on 1% whey protein concentrates, the deposition of protein was found to be 5 g/m<sup>2</sup> at the mouth of the tube. This number was found to vary between 0.1 g/m<sup>2</sup> to 15 g/m<sup>2</sup> depending on the Reynolds number and fluid temperature.

The fouling experienced in the food industry, for which the results were compared against, experiences a very high degree of fouling (Changani et al. 1997) and equipment is cleaned on a daily basis. This is much higher than a petrochem unit where cleaning might take place on an annual basis. Having comparable (to food industry) results indicate that fouling is an issue which needs attention.

Cleaning (in place) procedures (CIP) are an important part of the design of any heavily fouling systems. A preliminary evaluation of a caustic cleaning was done for the on-farm ethanol process (Tamhankar 2009). The process requires the circulation of a 2 -3 wt% caustic (NaOH) solution through the entire circuit for approximately four hours. The

spent caustic can be reused if the solids are removed from the solution by a simple filtration process. Packing compatibility, pre-heating, instrumentation and equipment selection for the process still need to be addressed. The fouling in the process also needs to be understood and evaluated in greater detail for the sweet sorghum processing standpoint.

#### VII.2 Fusel Alcohol Content:

The identification and quantification of fusel alcohols is of high importance in the alcoholic beverage manufacturing industry. This set of high boiling alcohols or fusels should be removed from the rectifying column to avoid possible accumulation and subsequent decrease in column efficiency (Muller et al. 1981). Among several higher alcohols identified as part of the group called fusels, isoamyl alcohol is found to be particularly abundant (Lea et al. 1995). Isobutyl alcohol and propanol are among other alcohols present in fermented beverages. For the identification and quantification of these compounds analyses have typically involved the use of gas chromatography (GC) (GE et al. 1971; Shawky M. Dagher 1975). Retention times and peak heights were compared with the standards of known concentrations to arrive at the quantitative results. Based on this information we used a GC coupled with a mass spectrograph (MS) to not only qualitatively establish what higher alcohols were in the system but also quantify the amounts present in the samples.

An Agilent 6890 N GC coupled with a 5973 MSD equipped with a DB-FFAP polyethylene glycol column was used for this analysis. The capillary column is a 20.0 m x 100  $\mu$ m x 0.20  $\mu$ m nitroterephthalic acid modified column and is specifically designed for applications involving organic acids, alcohols, aldehydes and ketones. The total run

time was 17 minutes. The temperature was 35 °C for the first three minutes, increased to 95 °C where it was held for the next 4 minutes, increased to 185 °C and then to 205 °C. The injection volume used was 0.5 µL. A split ratio of 20:1 was used to run the GC.

Standards from Absolute Standards Inc. were used to set up calibrations for fusel alcohol analysis. A three point calibration was set up with concentrations ranging from 0.025 g/L to 0.5 g/L. The components tested for are tabulated in Table 7.2.

Table 7.2: Calibration concentrations for fusel analysis						
	Absolute Standards Calibration Concentration				ion (g/L)	
Part #	Component	µg/ml	g/L	1	2	3
	Acetaldehyde	10000	10	0.050	0.10	0.50
	Ethyl acetate	10000	10	0.050	0.10	0.50
	Methanol	5000	5	0.025	0.05	0.25
	n-Propanol	10000	10	0.050	0.10	0.50
	Isobutyl alcohol (iso-Butanol)	10000	10	0.050	0.10	0.50
	3-Methyl-1-butanol	5000	5	0.025	0.05	0.25
92616	(S)-(-)-2-Methyl-1-butanol	5000	5	0.025	0.05	0.25

Table 7.9. Calibration concentrations for freed analysi

The samples to be tested had to be prepared before the GC/MS analysis. The main objective was to ensure the removal of solids from the broth before injection into the GC column. The samples were centrifuged in a microcentrifuge for 10 min at 13,000 g. After the centrifugation, the samples were filtered though a 13 mm diameter, 0.45 µm pore size Fischerbrand filter with the help of a 3 mL BD syringe, into a GC vial. A sample size of 1 ml is needed to safely carry out the analysis.

The analysis for fusel alcohols was done for 25 samples selected from a batch of 90 fermented samples available from the School of Biosystems and Agriculture Engineering. The samples included numerous samples from different time points in the fermentation, various varieties of sweet sorghum grown, juice extracted with and without leaves and plantings from several locations around Stillwater, OK. Table 7.3 gives the experimental results.

				(g/L)			
Sample No.	Samp	le Name		1-Butanol,3- methyl-	1-Propanol,1- methyl-	1-Butanol,3- methly-(S)	
10	10/9	EFAW	Small unstripped -1	0.06	0.04	0.01	
13	9/16	LCB	9-1	0.06	0.04	0.01	
17	9/16	LCB	14-2	0.05	0.03	0.01	
19	9/9	EFAW	M-81-1	0.07	0.02	0.01	
22	9/9	EFAW	H013-1	0.05	0.01	0.01	
24	9/9	EFAW	H019-1	0.06	0.01	0.02	
25	9/9	EFAW	Topper-1	0.07	0.01	0.02	

Table 7.3: Results for samples showing >0.05 g/L of isoamyl alcohol

Typical results from the table reflect quantities of fusel alcohol. Iso-amyl alcohols (3-methyl-1-butanol), the main constituent of fusel alcohols was found in almost all the samples, but in very small quantities. In samples where it was found the amount was less than 100 ppm. Iso butyl alcohol (2-methyl-1-propanol) was also found in trace quantities. Even though the quantities of the alcohols are low, accumulation over time can lower the efficiency of the column. The presence of fusel alcohols in the feed makes the presence of two draw points in the rectifying column important.

## CHAPTER VIII

#### CONCLUSIONS AND DISCUSSION

Observed in the early 1980s, studies wrote off the concept of "on-farm" ethanol due to unfavorable economic conditions. According to their investigation the return on the alcohol product, produced from corn, was very low for the operation of these plants. However, recent increase in fuel prices, technical advances in separation, and global interest in biofuels all provide encouragement towards investigating small scale ethanol again. And with the use of crops like sweet sorghum, the disadvantages faced with corn can be nullified.

As seen, there were attempts at setting up small scale ethanol production units in the 1980s. These studies identified several opportunities to improve farm scale operations. However, due to the decline in interest after the mid 1980s no significant progress in this field was made. Now, with the change in the global energy landscape and growing emphasis on biofuels, new approaches to deliver ethanol to the marketplace are needed. Decentralized, farm based production of ethanol is one such approach. However the viability of ethanol production from sweet sorghum, 'on-farm", depends heavily on the success of the separation and dewatering step.

At this point, technical advances in the field of distillation have offered an opportunity to re-examine distillation as a viable approach to on-farm ethanol separation. The inclusion of high efficiency trays, novel rectifier designs in association with Sulzer Chemtech, the use of a reboiler instead of boiler-steam and the use of an air-cooled heat exchanger provide potential cost savings and environmental benefits compared to previous attempts. All these results provide significant incentive to investigate the onfarm dewatering step and demonstrate it on a small scale. Conclusions for the research objectives based on our results have been presented in this section.

VIII.1 Ethanol dewatering technology selection:

Based on a technical evaluation of the myriad separation technologies available for ethanol water separation it was confirmed that distillation remains an appropriate technology to obtain ethanol (~95 wt %). For the final dewatering step, even though membrane technology supersedes adsorption based and solvent technologies in terms of energy use and ease of operation (Sander et al. 1991), it proves to be an expensive alternative technology, which is a key consideration for farm based processes. Molecular sieve adsorption achieved through temperature swing adsorption (TSA) is recommended for the final dewatering step. The combination of TSA with ethanol water distillation technology can perhaps offer an economical solution to on-farm ethanol dewatering.

#### VIII.2 On farm dewatering simulation:

The steady state simulation and the preliminary design indicate that on- farm ethanol is a viable proposition from an engineering standpoint. The simulations set up for on farm dewatering provide a steady state mass and energy balance for the system. The

system has a low energy usage as seen from the minimum energy simulations as well. Since it uses just 1% higher energy at steady state we can conclude that it is energy efficient in this layout in concept.

#### VIII.3 On-farm ethanol detailed design:

The detailed equipment designs based on the steady state on farm dewatering step have been proposed by industry leaders and veterans. The beer and rectifier columns both having a height of +30 ft are large enough to require installation in an outdoor location. However, unlike large commercial facilities, the OSU Bioenergy pilot plant is a small scale venture. It will use easily available resources such as air for the rectifier condenser, steam or natural gas combustion gases in the reboilers. Heat recovery and energy management are also important themes of this pilot plant. The stillage or the combustion gases are thus utilized to preheat the feed. And these are some features which provide novelty to the process. Even though similar recommendations were made in the past, little or no work has been pursued in this regard. The construction, demonstration and maintenance of the pilot plant will in the future answer several unanswered questions like the ones mentioned. Along with that, safety, maintenance and operation are some of the issues of on-farm ethanol dewatering which will be emphasized as the project progresses. Issues such as fouling, especially with respect to sweet sorghum processes have not been addressed in the past. On completion and subsequent operation of the process, data collected will shed light on the topics discussed.

The pilot plant when built will also feature state of the art instrumentation. This will help optimize process conditions for maximum ethanol output or low energy consumption or lowest cost. We realize that in reality a full fledged instrumentation

system might be very complicated or costly for farm applications. Thus future efforts will be guided towards simplifying the control and instrumentation strategy.

## VIII.4 Fusel Analysis:

The study conducted on the fusel alcohols in the on-farm feed confirmed the presence of higher alcohols. Though the quantities are small, approximately 100 ppm, more time needs to be devoted to finding out how the fusel alcohol concentration changes in the feed with time.

With a state-of-the-art demonstration unit, future studies can be geared towards data generation and analysis required for economic and technical optimization of an onfarm ethanol production facility. Results from this work could have significant economic impact on agriculture. The role of the farmer will expand from feedstock supplier to that of fuel producer and increase the economic opportunities for local communities.

## CHAPTER IX

#### RECOMMENDATIONS

The OSU Bioenergy Project's ethanol separation design has its roots in strong chemical engineering concepts. However, it must be understood that the design process for any chemical plant is a dynamic one. Thus details in this design capture only a snapshot of the dynamic changes. What we are attempting to do is to prepare ourselves for the changes that come along in the design. One way this has been done is the creation of detailed excel sheets where changes in decisions relating to the selection of equipment or instrumentation can be translated into for example changes in the number of valves, changes in the pump on a cell in the sheet. However, keeping in mind the present design picture we can make certain recommendations for the future direction the project should take.

- 1. One of the main concerns of the current detailed design is the estimated costs of the pilot plant envisioned. Clearly the break even analysis points to unfavorable economics. However, it should be understood, than unless the pilot plant is built and tests done to determine the economic feasibility we will never understand the 'true' minimum cost of the process. Having said that, even at this point there are several ways to reduce the capital cost associated with the plant:
  - a. A second look is needed for the process instrumentation in terms of design and economics. Based on current quotes the instrumentation accounts for

more than 40% of the capital cost. The current quotes include state of the art control applications like radar based level control. However, for a farm based operation one could use less expensive instrumentation and achieve the same results. The next step would be to look at other instrumentation options which can provide the same results but at lower costs.

- b. The size of the storage tanks is also an issue which needs quick resolution. One needs to look at the liquid inventory of the entire system and focus on the reconstitution philosophy to arrive at the correct sizes for storage tanks. If the process needs only 5000 gal tanks instead of 10,000 gal tanks we stand to save more than \$ 14,000. Also if the product and stillage are combined to produce a constant feed stream, it eliminates the need for an entire storage tank. Both scenarios need to be considered in the future.
- c. The use of 316 L stainless steel in the process is an added economic burden. The question of the use of 304 SS vs. 316L SS also remains unresolved. There might be some avenues in the process where cheaper alternative materials can be used and these options should be explored. Portions of the process thus need to be adaptive in terms of future experimentation. We can envision different sections of piping with different materials to understand the impact of corrosion and fouling.
- With energy conservation, recycling and sustainability becoming important issues one needs to give a thought to the application of these concepts to the OSU Bioenergy Pilot plant. These factors will also affect the economics and profitability of the project. The ideas of resource conservation, life cycle analysis,

industrial ecology and environmental sustainability can be applied to this process design and future project direction.

- 3. Other technical recommendations for the project are:
  - a. Study of the final dewatering step from 190 to 199+ proof and its inclusion into the process design to produce a fuel ethanol product.
  - b. Study of the fusel components in feed, stillage and product streams and its effects on the stability of the over time.
  - c. Simplification of the process to ensure operatability by farmers.

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# APPENDIX A1

# CHEMCAD 6.1.3: OSU Bioenergy Output

The following sections (Appendix A1 and A2) include detailed CHEMCAD outputs for the OSU Bioenergy pilot plant. **Appendix A1 has results for operation of the beer still with a reboiler**. The combuster has been modeled as a Gibb's reactor with methane as fuel. Combustion gases provide heat to vaporize beer still bottoms for the required vapor rate.



Fig A1.1: Chemcad flowsheet for OSU alternative design with beer column reboiler and air-cooled condenser

## CHEMCAD 6.1.3

Job Name: OSU\_Bioenergy\_reboiler\_E1\_sensitivity Date: 11/24/2009 Time: 11:30:25

## FLOWSHEET SUMMARY

Equipment		Label	Stream		Numbers		
1	SCDS		19	30	-17	-9	
2	SCDS		27	17	-20	-18	
3	HTXR		1	21	-8	-2	
5	REAC		б	-7			
б	MIXE		4	5	-б		
8	HTXR		15	7	-16	-13	
9	DIVI		22	-15	-1		
10	MIXE		23	2	-19		
11	PUMP		11	-21			
12	PUMP		9	-22			
13	PUMP		18	-23			
14	HTXR		20	-24			
15	DVSL		24	-29	-25		
16	PUMP		25	-26			
17	DIVI		26	-28	-27		
18	VALV		16	-30			

## Stream Connections

Stream	Equ	ipme	nt	Stream		Equipment		S	tream	n				
Equipment														
Fr	om	То				Fro	om	То				From	Т	0
1	9	3			13		8			23		13	1	0
2	3	10			15		9	8		24		14	1	5
4		6			16		8	18		25		15	1	б
5		6			17		1	2		26		16	1	7
6	6	5			18		2	13		27		17		2
7	5	8			19	-	LO	1		28		17		
8	3			:	20		2	14		29		15		
9	1	12		:	21	-	11	3		30		18		1
11		11		:	22	-	12	9						
Calculatio Flash algo	n mc rith	ode : nm :	Seo Noi	quen cmal	tial									
Equipment	Calc	ulat	ion	Seq	uenc	е								
6 11	5	1	2	14	15	16	17	13	12	9	8	18	3	10
Equipment 1	Recy	rcle	Sequ	lenc	е									
1 2	14	15	16	17	13	12	9	8	18	3	10			
Recycle Cu 19 30	t St 27	ream	.s	atho	d •	Dire	at (	Subat	· + · · + ·	n				

Max. loop iterations 40

## CHEMCAD 6.1.3

Job Name: OSU\_Bioenergy\_reboiler\_E1\_sensitivity Date: 11/24/2009 Time: 11:30:25

Recycle Convergence Tolerance

Flow rate	1.000E-003
Temperature	1.000E-003
Pressure	1.000E-003
Enthalpy	1.000E-003
Vapor frac.	1.000E-003

Recycle calculation has converged.

#### CHEMCAD 6.1.3

Page 3

Job Name: OSU\_Bioenergy\_reboiler\_E1\_sensitivity Date: 11/24/2009 Time: 11:30:26

Overall Mass Balance	lbmol	L/h	lb/h			
	Input	Output	Input	Output		
Ethanol	2.351	2.355	108.321	108.475		
Water	107.334	112.068	1933.628	2018.896		
Lactic Acid	0.068	0.068	6.167	6.167		
Glycerol	0.100	0.100	9.240	9.239		
Acetic Acid	0.070	0.070	4.179	4.179		
Succinic Acid	0.005	0.005	0.586	0.586		
Methane	2.369	0.000	38.000	0.000		
Carbon Dioxide	0.000	2.369	0.000	104.244		
Nitrogen	128.346	128.346	3595.480	3595.480		
Oxygen	34.117	29.380	1091.720	940.132		
Total	274.761	274.760	6787.320	6787.397		
CHEMCAD 6.1.3				Page 4		

Job Name: OSU\_Bioenergy\_reboiler\_E1\_sensitivity Date: 11/24/2009 Time: 11:30:26

#### COMPONENTS

	ID #	Name	Formula
1	134	Ethanol	С2Н6О
2	62	Water	Н2О
3	680	Lactic Acid	С3Н6ОЗ
4	268	Glycerol	С3Н8ОЗ
5	130	Acetic Acid	C2H4O2
б	277	Succinic Acid	С4нбО4
7	2	Methane	CH4
8	49	Carbon Dioxide	CO2
9	46	Nitrogen	N2
10	47	Oxygen	02

#### THERMODYNAMICS

K-value model : NRTL Vapor Phase Association Enthalpy model : Latent Heat Liquid density : Library Std vapor rate reference temperature is 60 F. Atmospheric pressure is 14.6959 psi. NRTL Parameters: Tij = Aij + Bij/T + Cij \* Ln(T) + Dij \* T (T Deg K) I J Bij Bji Alpha Aij Aji Cij Cji Dij Dji 1 2 -55.17 670.44 0.303 0.00 0.00 0.00 0.00 0.00 0.00 1 4 398.44 79.51 0.296 0.00 0.00 0.00 0.00 0.00 0.00 1 5 -147.79 105.31 0.299 0.00 0.00 0.00 0.00 0.00 0.00 2 258.11 -274.35 1.011 0.00 0.00 0.00 0.00 0.00 0.00 4 2 5 424.02 -110.57 0.300 0.00 0.00 0.00 0.00 0.00 0.00

Warning : BIP matrix is less than 50 % full.

#### CHEMCAD 6.1.3

Job Name: OSU\_Bioenergy\_reboiler\_E1\_sensitivity Date: 11/24/2009 Time: 11:30:26 EQUIPMENT SUMMARIES

Page 5

#### Scds Rigorous Distillation Summary

Equip. No.	1	2
Name		
No. of stages	13	13
1st feed stage	1	1
2nd feed stage	13	13
Condenser spec		0.9000
Cond comp i pos.	0	1
Cond press drop psi	1.3000	
Colm press drop psi		2.4600
Est. dist. rate	13.3000	2.7500
(lbmol/h)		
Est. reflux rate	121.0000	11.0000
(lbmol/h)		
Est. T top F	202.0000	173.0000
Est. T bottom F	214.0000	212.0000
Top pressure psia	18.0000	15.3000
Calc Reflux mole	139.7946	21.1374
(lbmol/h)		
Calc Reflux mass lb/h	2936.4609	888.6635
Calc. tolerance	0.0002	8.9713e-007

## Heat Exchanger Summary

Equip. No.	3	8	14
Name			
1st Stream dp psi	0.1150		0.5900
2nd Stream dp psi	0.0100		
2nd Stream T Out F	120.0000		
1st Stream VF Out		0.0130	1.0000e-007
No. of SS Passes	0	1	0
No. of TS Passes	0	1	0
Calc Ht Duty MMBtu/h	0.1197	0.5973	-0.3630
LMTD (End points) F	100.7581	211.3572	
LMTD Corr Factor	1.0000	1.0000	1.0000
lst Stream Pout psia	19.8850	20.0000	14.7100
2nd Stream Pout psia	39.1300	40.0000	

## Reactor Summary

Equip. No.	5
Name	
Thermal mode	1
Temperature F	771.4632
Key Component	7
Frac. Conversion	1.0000
Calc H of Reac.	-344929.8750
(Btu/lbmol)	

# APPENDIX A2

# CHEMCAD 6.1.3: Steam Case Simulation Output

The detailed CHEMCAD output for operation of the OSU Bioenergy Pilot Plant **with stripping steam introduced in the Beer still** is given here. Steam is introduced into the beer still at 250 lb/h. Feed introduced in both cases remains the same.



Fig A2.1: Chemcad flowsheet for the traditional design with stripping steam in beer column

```
CHEMCAD 6.1.3
```

Page 1

```
Job Name: OSU_Bioenergy_steam-E1_sensitivity Date: 12/01/2009
Time: 10:24:02
FLOWSHEET SUMMARY
Equipment
            Label
                           Stream Numbers
      SCDS
                           11
                                6 -12
                                      -4
   1
   2
                           20
                               12 -13 -10
      SCDS
                                   -5 -3
   3 HTXR
                           16
                               14
   4
      CONT
                            2
                               -б
   5
      PUMP
                            1 -14
```

Equipment		Label	Sti	ream	Numbers
б	PUMP		10	-15	
8	MIXE		15	3	-11
9	PUMP		4	-16	
10	HTXR		13	-17	
11	DVSL		17	-22	-18
12	PUMP		18	-19	
13	DIVI		19	-21	-20

#### Stream Connections

Stream	tream Equipment		Stream	Equip	ment	Stream	Equip	ment
	From	То		From	То		From	То
1		5	11	8	1	18	11	12
2		4	12	1	2	19	12	13
3	3	8	13	2	10	20	13	2
4	1	9	14	5	3	21	13	
5	3		15	б	8	22	11	
б	4	1	16	9	3			
10	2	б	17	10	11			

Calculation mode : Sequential Flash algorithm : Normal

Equipment Calculation Sequence 4 5 1 2 10 11 12 13 6 9 3 8

Equipment Recycle Sequence 1 2 10 11 12 13 6 9 3 8

Recycle Cut Streams 11 20

Recycle Convergence Method: Direct Substitution Max. loop iterations 40

Recycle Convergence Tolerance Flow rate 1.000E-003 Temperature 1.000E-003 Pressure 1.000E-003

#### CHEMCAD 6.1.3

Job Name: OSU\_Bioenergy\_steam-E1\_sensitivity Date: 12/01/2009 Time: 10:24:02

Enthalpy	1.000E-003
Vapor frac.	1.000E-003

Recycle calculation has converged.

Page 2

## CHEMCAD 6.1.3

Time: 10:24:02

•••••••••••••••••••••••••••••••••••••••		/ ==					
	Input	Output	Input	Output			
Ethanol	2.344	2.345	107.989	108.020			
Water	138.348	138.348	2492.341	2492.341			
Lactic Acid	0.068	0.068	6.168	6.168			
Glycerol	0.100	0.100	9.242	9.242			
Acetic Acid	0.070	0.070	4.180	4.180			
Succinic Acid	0.005	0.005	0.586	0.586			
Total	140.936	140.936	2620.506	2620.537			

## CHEMCAD 6.1.3

Job Name: OSU\_Bioenergy\_steam-E1\_sensitivity Date: 12/01/2009 Time: 10:24:02

#### COMPONENTS

	ID #	Name	Formula
1	134	Ethanol	C2H6O
2	62	Water	H2O
3	680	Lactic Acid	С3н6О3
4	268	Glycerol	С3Н8ОЗ
5	130	Acetic Acid	C2H4O2
б	277	Succinic Acid	C4H6O4

#### THERMODYNAMICS

K-value model	:	NRTL
		Vapor Phase Association
Enthalpy model	:	Latent Heat
Liquid density	:	Library

Std vapor rate reference temperature is 60 F. Atmospheric pressure is 14.6959 psi.

NRTL Parameters: Tij = Aij + Bij/T + Cij \* Ln(T) + Dij \* T (T Deg K)

I	J	Bij	Bji	Alpha	Aij	Aji	Cij	Cji	Dij	Dji
1	2	-55.17	670.44	0.303	0.00	0.00	0.00	0.00	0.00	0.00
1	4	398.44	79.51	0.296	0.00	0.00	0.00	0.00	0.00	0.00
1	5	-147.79	105.31	0.299	0.00	0.00	0.00	0.00	0.00	0.00
2	4	258.11	-274.35	1.011	0.00	0.00	0.00	0.00	0.00	0.00
2	5	424.02	-110.57	0.300	0.00	0.00	0.00	0.00	0.00	0.00

Warning : BIP matrix is less than 50 % full.

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#### Page 4

# Page 3

Job Name: OSU\_Bioenergy\_steam-E1\_sensitivity Date: 12/01/2009 Time: 10:24:03 EQUIPMENT SUMMARIES

Scds Rigorous Distillation Summary

Equip. No.	1	2
Name		
No. of stages	13	13
1st feed stage	1	1
2nd feed stage	13	13
Condenser spec		0.9380
Cond comp i pos.	0	1
Reboiler spec.	0.0100	
Reboiler comp i	1	0
Colm press drop psi	1.3000	2.4600
Est. dist. rate	13.3000	2.7500
(lbmol/h)		
Est. reflux rate	121.0000	11.0000
(lbmol/h)		
Est. T top F	202.0000	173.0000
Est. T bottom F	214.0000	212.0000
Top pressure psia	18.0000	15.3000
Calc Reflux mole	137.2323	21.9713
(lbmol/h)		
Calc Reflux mass lb/h	2844.4639	923.9875
Calc. tolerance	1.2701e-005	9.6706e-007

# APPENDIX B

## Sulzer Chemtech Column Design Data

The design of the beer still and rectifier is based on the following simulation. The simulation has been run on ProII software. Both columns have been set up as a single column for convergence ease.



Fig B1: Pro II Output for on farm simulation

Based on the simulation above, Dan Summers from Sulzer Chemtech has summarized the design of the two columns in the following diagram (Daniel R. Summers 2008). The design of the beer and rectifier is based on the following simulation. The simulation has

# been run on ProII software. Both columns have been set up as a single column for

convergence ease.



Fig B.2: Column Design: (a) Rectifier detailed dimensioned drawing (b) Beer Still design



Fig B.2: Column Design: Detailed dimensioned drawing for Beer Still design

# APPENDIX C1

## Reboiler Heat Exchanger Design

Design Basis for the Reboiler:

The reboiler design was completed by Dr. Ken Bell (Bell 2009). The data provided for the design was drawn out of the simulations. The following information was provided to initiate the reboiler design.

- Pressure at the bottom of the beer column is 16.3 psia.
- The temperature of the liquid leaving the column will be  $217.3 \,^{\circ}$ F.
- The heat duty of the reboiler will be 0.319 MMBtu/h.
- The vapor generation rate for the reboilers is fixed at 330 lb/hr. The rate of liquid leaving the bottoms of the beer column is 1,864 lb/hr.

Some numbers from the notes:

Vapor delivery rate is 330 lb/hr. At 16.3 psia,

$$T_{sat} = 217.3 \text{ °F}$$

$$h_{fg} = 966 \text{ Btu/lb}$$

$$Q = 330 \frac{\text{lb}}{\text{hr}} \times 966 \frac{\text{Btu}}{\text{lb}} = 319,000 \text{ Btu/hr}$$

$$Q_{design} = 350,000 \text{ Btu/hr}$$

$$\rho_v = \frac{1}{24.35} \frac{\text{ft}^3}{\text{lb}} = 0.0411 \text{ lb/ft}^3$$

$$\rho_l = \frac{1}{0.01675} \frac{\text{ft}^3}{\text{lb}} = 59.7 \text{ lb/ft}^3$$

$$C_{pl} = 1.004 \frac{\text{Btu}}{\text{lb}} \text{ °F}$$

Heat transfer area estimates:

We assume a low carbon steel <sup>3</sup>/<sub>4</sub> inch OD for the tubes. The walls are 0.065 inch thick and the ID of the tubes is 0.62 inch. To calculate the overall heat transfer coefficient assume flue gases in the shell side of the heat exchanger and stillage (almost water) in the tube side of the heat exchanger.

$$h_{flue gases} = 20 Btu/hr ft^2 °F$$
  
 $r_{flue gases} = 0.005 hr ft^2 °F/Btu$ 

$$r_{wall} \approx \frac{0.065}{12 \times 30} = 0.0002 \text{ hr ft}^{2} \text{ F/}_{Btu}$$

$$r_{water} = 0.005 \text{ hr ft}^{2} \text{ eV}/Btu$$

$$h_{water} = 1500 \text{ Btu}/_{hr} \text{ ft}^2 \text{ °F}$$
$$U_o = \frac{1}{\frac{1}{20} + 0.0005 + 0.0002 + (0.005 + \frac{1}{1500}) \times (\frac{0.75}{0.62})} = 17.4 \text{ Btu}/_{hr} \text{ ft}^2$$

The value of heat transfer coefficient will be used for the first design pass. It will be changed later on, if required. Assume that the inlet flue gas is 600 °F and the exit temperature is 300 °F. The LMTD can then be calculated by the following equation.



Fig C.2: Representation of LMTD for reboiler

LMTD = 
$$\frac{(600-217.3) \cdot (300-217.3) \circ F}{\ln(\frac{600-217.3}{300-217.3}) \circ F} = 195.8 \circ F$$

The overall area required for the heat exchanger is then calculated using the following equation as the configuration will be pure counterflow.

$$A_{o} = \frac{Q}{U_{o} \times LMTD} = \frac{350,000 \text{ Btu/}_{hr}}{17.4 \text{ Btu/}_{hr} \text{ ft}^{2} \times 195.8} = 97.3 \text{ ft}^{2}$$

Next, the flow rate required is determined as a function of exit quality or vapor fraction of the exiting stream.

Entering liquid flow rate= 
$$\frac{330 \text{ lb vapor/hr}}{\text{Fractional vaporization}}$$

The volumetric flow rate in gallons per minute is calculated by the following equation.

volumetric flow rate gpm= 
$$\frac{\text{volumetric flow rate ft}^3 \times 7.48 \frac{\text{gal}}{\text{ft}^3}}{60 \frac{\text{min}}{\text{hr}}}$$

Table C1 tabulates the volumetric flow rates in gallons per minute required for a fractional vaporization range of 1% to 5%. The heat exchanger design will be based on

the exit quality of 1% vapor.

Fractional vaporization	Flow rate (lb/hr)	Volumetric flow rate (ft <sup>3</sup> /hr)	Volumetric flow rate (gpm)
0.01	33,000	553	69.0
0.02	16,500	276	34.5
0.03	11,000	184	23.0
0.04	8,250	138	17.3
0.05	6,600	111	13.8

Table C.1: Flow rate required as a function of exit quality

For a design inlet velocity of 6 ft/s the number of tubes in parallel is

$$N_{\text{tubes}} = \frac{\text{volumetric flow rate ft}^3/h}{\left(6\frac{\text{ft}}{\text{s}}\right) \times \left(3600\frac{\text{s}}{\text{min}}\right) \times \left(\frac{\pi}{4}\right) \times \left(\frac{0.620}{12}\text{ ft}\right)^2}$$

For 1% vaporization the number of tubes calculated is 12.2 tubes. The tube length is calculated from the following equation

Tube length= 
$$\frac{97.3 \text{ ft}^2}{12.2 \text{ tubes} \times \pi \times \frac{0.75 \text{ in}}{12 \text{ ft}}} = 41.3 \text{ ft/tube}$$

Since the velocity of 6 ft/s yields an excessively high tube length, we assume a velocity 3 ft/s to find out the number of tubes. Also, consider 5/8 inch OD tube instead of a <sup>3</sup>/<sub>4</sub> inch tube. The ID of the tube is 0.495 inch. The number of tubes is calculated using the previous equation to be 38.4 and the length of the tube is calculated to be 15.6 ft. This value is still high. If we consider finned tubes of <sup>3</sup>/<sub>4</sub> inch which has16 fins/inch, the length of tubes can be brought down further. The fin height is 0.065 inch and the fin thickness is 0.065 inch.

$$\frac{A_{o}}{L} = 16 \times 12 \times \frac{\pi}{4} \times \left[ (0.75)^{2} - (0.625)^{2} \right] \times \frac{1}{144} \times 2$$
$$+ \left[ \frac{12 - 16 \times (0.15) \times 12}{12} \right] \times \pi \times \frac{0.625}{12} = 0.48 \text{ ft}^{2}/\text{ft}$$

The outside effective heat transfer are per foot is much larger than the outside tube area and is calculated to be  $0.48 \text{ ft}^2/\text{ft}$ . This reduces the length of the tube needed to 5.3 ft.

# Tube Layout:

The tubes can be placed in an equilateral triangle layout of 1.5 pitch ratio or 1.125 pitch.



Fig C3: Tube layout for reboiler

To accommodate the tubes an 8 inch pipe will be used as a shell. The standard ID for this

pipe is 7.981 inch. There will be 37 tubes in the tube bundle.

Shell side heat transfer:

Information needed to calculate the volumetric flowrate is given below.

At 1 atm and 600 °F,

$$\rho_{air} = 0.038 \text{ lb/ft}^3$$

At 1 atm and 300 °F,

$$\rho_{air} = 0.051 \text{ lb/ft}^3$$

At 1 atm and the average temperature 450 °F,

$$\rho_{air} = 0.042 \text{ lb/ft}^{3}$$

$$C_{p,air} = 0.245 \text{ Btu/(lb °F)}$$

$$\mu_{air} = 18 \times 10^{-6} \frac{\text{lb}}{\text{s ft}} = 6.48 \times 10^{-2} \text{lb/(ft hr)}$$

$$k_{air} = 23.5 \times 10^{-3} \frac{\text{Btu}}{\text{hr ft °F}}$$

$$Pr = \frac{0.254 \text{ Btu/(lb °F) x } 6.48 \times 10^{-2} \text{lb/(ft hr)}}{2.35 \times 10^{-2} \frac{\text{Btu}}{\text{hr ft °F}}} = 0.676$$

$$\dot{w}_{air} = \frac{350,000 \text{ Btu}}{0.245 \text{ Btu}} = 4,716 \text{ lb/hr}$$

$$\dot{v_{air}} = \frac{4.176 \text{ lb/hr}}{0.042 \text{ lb/ft}^3} = 1.13 \times 10^5 \frac{\text{ft}^3}{\text{hr}} = 31.5 \text{ ft}^3/\text{s}$$

The maximum allowable flow space in the shell

Max flow space = 
$$7.981$$
 in- $(7 \times 0.75)$ in =  $2.731$  in

$$A_{max} = \frac{2.731 \text{ in} \times 8 \text{ in}}{(12 \text{ in/}{ft})^2} = 0.151 \text{ ft}^2$$
$$\frac{31.5 \text{ ft}^3/\text{s}}{\text{V}_{max}} = \frac{31.5 \text{ ft}^3/\text{s}}{0.151 \text{ ft}^2} = 208 \text{ ft/s}$$

The calculated heat transfer coefficient

$$h_{i} = 0.0061 \times 0.245 \text{ Btu/(lb °F)} \times \frac{4.716 \text{ lb/hr}}{0.151 \text{ ft}^{2}} \times (\frac{1}{0.676})^{2/3} \times (\frac{6.48}{5.47})^{0.14} = 54.5 \frac{\text{Btu}}{\text{hr ft}^{2} \text{°F}}$$

The pressure drop calculated across one baffle space for 7 rows is

$$\Delta p_{b,i} = \frac{4 \times 0.12 \times (4,716)^2 \times 7 \times (\frac{1}{1.024})}{2 \times 0.042 \times (4.17 \times 10^8) \times (0.151)^2} = 91.4 \text{ lb/ft}^2$$

Taking a correction factor of 20 lb/ft<sup>2</sup> the pressure drop is calculated to be 0.13 lb/inch<sup>2</sup>. In the above calculations the shell side heat transfer coefficient is calculated to be 54.5 Btu/ hr ft<sup>2</sup> °F, which is extremely high. The calculations are repeated with a lower heat transfer coefficient,  $h_o = 30$  Btu/ hr ft<sup>2</sup> °F. In the second pass the results were

$$U_0 = 24.5 \text{ Btu/}_{hr ft^2}$$
  
 $A_0 = 73 \text{ ft}^2$ 

$$L = \frac{73 \text{ ft}^2}{(3 \text{ tubes}) \times (0.484 \text{ ft}^2/\text{ft})} = 4.08 \text{ ft tube length}$$

Summary notes by Dr. Ken Bell:

"We can keep the basic tube layout, reduce tube length and use double sequestered baffles (Not optimal but reasonable).

Shell: 8 inch standard pipe, 4 ft tube length TEMA AEL. 8 inch baffle spacing, double segmented baffles. Shell side expansion joint/roll required.

Tube: Stainless steel, <sup>3</sup>/<sub>4</sub> inch OD. Finned, 16 fins/inch. Fin height 1/16 in, Tube ID 0.495 inch.



Fig C.1: Schematic of bioethanol reboiler

Need to determine whether these tubes are economically available. Alternatively, low fin titanium tubes with 30-40 fins per inch are available and would be acceptable. The design needs to be checked for vibrations, mechanical code requirements and optimization of thermal hydraulic characteristics.

# APPENDIX C2

# Condenser: Heat Exchanger Design

The design of the air-cooled heat exchanger was undertaken by Dr. Ken Bell as well (Bell 2008).

Design Basis:

The heat exchanger is to condense 447.76 lb/hr of reflux and 110.77 lb/hr of ethanol product (90 wt% ethanol and 10 wt% water) at 173.1 °F. The condenser is designed as an air cooled heat exchanger operating at atmospheric pressure using air at 100 °F. Assuming a latent heat of condensation to be 413 Btu/lb and neglecting subcooling, the heat duty is calculated to be

Q=558.53 
$$\frac{lb}{hr} \times \frac{413Btu}{lb} = 231,000 \text{ Btu/hr}$$

The design outlet temperature is taken as 140 °F. The LMTD is calculated as

LMTD=
$$\frac{(173.1-100)^{\circ}F-(173.1-140)^{\circ}F}{\ln(\frac{3.1}{33.1})^{\circ}F} = 50.5 ^{\circ}F$$

The air flow rate required through the air cooled heat exchanger

$$\dot{\text{m}}_{\text{air}} = \frac{231,000 \text{Btu/hr}}{\left(0.24 \frac{\text{Btu}}{\text{lb}^{\circ}\text{F}}\right) \times (40^{\circ}\text{F})} = 24,100 \text{ lb/hr}$$

$$\dot{v}_{air} = \frac{24,100 \text{ lb/hr}}{0.069 \text{ lb/ft}^3} = 349,300 \frac{\text{ft}^3}{\text{hr}} = 97.0 \text{ ft}^3/\text{s}$$

The face velocity needed is calculated for a range of velocities starting from 6 ft/s and is given in Table C2.1.

Table C2.1: Face area calculations					
	<i>v<sub>air</sub></i> , ft/s	$A_{face}, \operatorname{ft}^2$			
	10	9.7			
	9	10.8			
	8	12.1			
	6	16.2			

Tube configuration:

The base tube configuration is of a 1 inch OD x 0.866 in ID of 14 BWG wall made of steel. The tube is assumed to have 10 fins per inch. These fins are  $\frac{1}{2}$  inch high with an 0.015 inch mean thickness and are made of aluminum. The tubes will be arranged in an equilateral triangular pitch of 2.187 inch.

The fin area per tube

$$A'_{fin} = \left(10 \ \frac{fins}{inch}\right) \times \left(12 \ \frac{inch}{ft}\right) \times \frac{\pi}{4} \left[\frac{2 \ in^2 - 1 \ in^2}{144 \ \frac{in^2}{ft^2}}\right] \times 2 = 3.93 \ \frac{ft^2}{ft} \text{ of tube}$$

Bare tube area per ft of tube

$$A_{bare} = \pi \times \left(\frac{1 \text{ inch}}{12 \frac{\text{in}}{\text{ft}}}\right) \times \left(1 - \frac{\left(120 \frac{\text{fin}}{\text{ft}}\right) \times (0.015 \text{ in})}{12 \frac{\text{in}}{\text{ft}}}\right) = 0.23 \text{ ft}^2/\text{ft}$$

Assuming a fin efficient of 0.90

$$A'_{eff} = 0.23 \frac{ft^2}{ft} + 3.93 \frac{ft^2}{ft} \times 0.9 = 3.77 \frac{ft^2}{ft} \text{ of tube}$$

Tube layout:

The tubes are assumed to have 2.00 inch OD and their layout is shown in Fig C2.1.



Fig C2.1: Tube layout for the air cooled condenser

Free flow area between two adjacent tubes is calculated

$$A'_{\frac{\text{free}}{\text{ft of tube}}} = \frac{(12 \text{ in}) \times (2.1875 \text{ in-1 in}) - 120 \times (1 \text{ in}) \times (0.015 \text{ in})}{144 (\text{in/ft})^2} = 0.0865 \frac{\text{ft}^2}{\text{ft}} \text{ ot tube}$$

The face area per foot of tube is

$$A'_{face} = \frac{2.1875 \text{ in} \times 12 \text{ in}}{144 \left(\frac{\text{in}}{\text{ft}}\right)^2} = 0.1823 \frac{\text{ft}^2}{\text{ft}} \text{ of tube}$$

Conversion between face velocity and  $v_{max}$ , the maximum velocity achieved by the air, as it flows through the tube field

$$v_{max} = v_{air} \times \frac{0.1823}{0.0865} = 2.11 v_{air}$$

Calculation of air side heat transfer coefficient by the Briggs and Young correlation (Kraus et al. 2001)

$$\frac{h_o D_r}{k} = 0.134 \left(\frac{D_r \rho v_{max}}{\mu}\right)^2 P r^{1/3} \left(\frac{H}{s}\right)^{-0.2} \left(\frac{Y}{s}\right)^{-0.12}$$

Where,  $D_r = 1.00$  inch, the root diameter of the fins

H = 0.50 inch, height of the fin

Y = 0.015 inch, thickness of the fin

s = 0.100 - 0.015 = 0.85 inch, spacing between the fins

Air properties at 120 °F are, k = 0.016

$$\rho = 0.068 \text{ lb/ft}^3$$
  
 $\mu = 13 \times 10^{-6} \text{ lb/s ft} = 0.0465 \text{ lb/ft hr}$   
 $Pr = 0.7$ 

The value of  $h_o$  calculated using the Briggs and Young correlation is

$$h_0 = 4.68 \times 10^{-3} (\frac{v_{max} ft}{hr})^{0.68}$$

Calculation of air side coefficient based on the maximum air velocity is shown in the Table C2.2. Here,  $h_{air}$  is based on the effective total air side (finned) heat transfer area of 3.77 ft<sup>2</sup>/ft of tube.

v <sub>face</sub> ft/s	v <sub>max</sub> ft/s	v <sub>max</sub> ft/hr	<i>h<sub>air</sub></i> Btu/hr ft <sup>2</sup> °F
12	25.3	$9.12 \times 10^4$	11.0
10	21.1	$7.60 \ge 10^4$	9.75
9	19.0	6.84 x 10 <sup>4</sup>	9.08
8	16.9	6.08 x 10 <sup>4</sup>	8.38
6	12.7	$4.56 \ge 10^4$	6.89

Table C2.2: Variation of air side heat transfer coefficient

Condensing heat transfer coefficient:

We must consider whether the condensing coefficient is controlled by gravity drainage of the condensate film or by the vapor shear driving the condensate film in the turbulent annular flow regime. In either case we must make some estimate of the number and length of the tubes. To do so, we make a rough estimate of the area required. For this purpose, our estimate for the overall heat transfer coefficient based on the effective outside tube area is 6 Btu/hr ft<sup>2</sup>  $^{\circ}$ F.

Then,

$$A_{eff, est.} = \frac{231,000 \text{ Btu/hr}}{(50.5 \text{ }^{\circ}\text{F}) \times (6\frac{\text{Btu}}{\text{hr ft}^{2} \text{ }^{\circ}\text{F}})} = 762 \text{ ft}^{2}$$

The number of tubes required is

No. of tubes=
$$\frac{762 \text{ ft}^2}{3.77 \text{ ft}^2/\text{ft}} = 202 \text{ ft}$$

This requirement is satisfied by the following combinations

Table C2.3: No. of tubes for condenser

Tube length	No. of tubes
ft	
4	50
5	40
6	34
8	25

We can fit 5.5 tubes side by side in a row that is 1 foot wide.



Fig C2.1: Tube arrangement in a row

A three row configuration would look like



Fig C2.2: Tube configuration

A possible configuration would be 3 rows of 4 ft long tubes with 2 rows of 17 tubes each and one row of 16 tubes. This would require a tube bank width of  $17 \times (2.1875 \text{ inch}) =$ 

37.2 inch + side structure or, roughly a 3 ft by 4 ft face area and a unit face dimension of 3.5 ft wide and 5 ft long. All tubes could be in parallel, with two headers, facilitating cleaning. Based on the tube layout, the condensing coefficients can be calculated. Gravity controlled: Kerr correlation (Logan et al. 2002)

$$h_{c} = 0.761 \left[ \frac{k_{l}^{3} \rho_{L} (\rho_{L} - \rho_{v}) gL}{W_{T} \ \mu_{L}} \right]^{1/3}$$

Where  $W_T$  is the mass condensed per tube and L is the length of the tube. For our configuration,

$$W_{T} = \frac{558.3 \text{ lb/hr}}{50 \text{ tubes}} = 11.2 \frac{\text{lb}}{(\text{hr tube})}$$

And, L = 4 ft

From Dr. Bell's notes

 $k_L = 0.0896$  Btu/ hr ft<sup>2</sup> °F  $\mu_L = 1.02$  lb/ft hr  $\rho_L = 47.3$  lb/ft<sup>3</sup>  $\rho_v = 0.102$  lb/ft<sup>3</sup>

The gravity controlled coefficient is calculated to be 469 Btu/hr ft<sup>2</sup>  $^{\circ}$ F.

For the calculation of vapor shear controlled coefficient calculation the Boyko Kruzikni correlation (Perry et al. 2007) is used for total condensation

$$h_{c} = 0.024 \frac{k_{L}}{D_{i}} \left(\frac{D_{i}G_{T}}{\mu_{L}}\right)^{0.8} Pr_{L}^{0.43} \left[\frac{1 + \sqrt{\frac{\rho_{L}}{\rho_{v}}}}{2}\right]$$

Where,

$$G_{\rm T} = \frac{\text{Mass condensed}}{\text{Flow cross section}}$$

Flow cross section assuming 50 tubes in parallel

$$s_{\text{flow}} = 50 \times \frac{\pi}{4} \times \left(\frac{0.866}{12}\right)^2 = 0.205 \text{ ft}^2$$

The value of  $h_c$  calculated using the vapor shear controlled regime is 21.5 Btu/ hr ft<sup>2</sup> °F. Under this regime, the Reynold's number was calculated to be 193, suggesting laminar flow, suggesting vapor shear flow is not likely to be the controlling mechanism. Thus, assuming condensation is gravity controlled and  $h_c$  is 469 Btu/ hr ft<sup>2</sup> °F, the overall heat transfer coefficient is calculated. Assuming  $v_{face}$  of 8 ft/s,  $U_{A,eff}$  is calculated to be 5.72 Btu/ hr ft<sup>2</sup> °F.

$$U_{\text{Aeff}} = \frac{1}{\frac{1}{8.38} + \frac{(0.067)(3.77)}{12(28)\pi(\frac{0.866}{12})} + \frac{3.77}{469(\pi)(\frac{0.866}{12})} + \frac{0.001(3.77)}{\pi(\frac{0.866}{12})}} = 5.72 \text{ Btu/ hr ft}^{2\circ F}$$

This is within calculation uncertainty to the assumed overall heat transfer coefficient. The layout of the heat exchanger will resemble Fig C2.4 and will have 3 rows of tubes (17-16-17) totaling 50 tubes.



Fig C2.4: Air cooled condenser schematic

There will be a 30 inch diameter fan and a motor between  $\frac{1}{2}$  and  $\frac{3}{4}$  HP.

Air side Pressure Drop:

Calculation of the air side pressure drop will be considered for flow rate 24,100 lb/hr and an additional correction factor of 10% to compensate for calculation discrepancies and 6% safety factor.

For the tube bank, the friction loss is given by the Robinson Briggs (McKetta 1984) equation

For this the coefficient is calculated

$$f_r = 18.93 \left(\frac{D_r \rho v_{max}}{\mu}\right)^{-0.316} \left(\frac{P_t}{D_r}\right)^{-0.927}$$

In the above equation

$$\operatorname{Re} = \left(\frac{D_{r}\rho v_{max}}{\mu}\right) = \left(\frac{\left(\frac{1.00 \text{ in}}{12}\right) \left(\frac{0.068 \text{ lb}}{\text{ft}^{3}}\right) \left(6.63 \times 10^{4} \frac{\text{ft}}{\text{hr}}\right)}{0.0468 \frac{\text{lb}}{\text{ft} \text{ hr}}}\right) = 8030$$

For a mass air flow rate of 24,100 lb/hr,

$$v_{max} = \frac{1.1 \times \left(24,100 \frac{\text{lb}}{\text{hr}}\right) \times 2.11}{0.068 \frac{\text{lb}}{\text{ft}^3} \times 4 \text{ft} \times 3.1 \text{ ft}} = 18.4 \text{ ft/s}$$

# From the Robinson Briggs equation friction loss coefficient was calculated to be 0.535.

The pressure drop across is given by,

$$\Delta p = \frac{f_r N \rho v_{max}^2}{g_c} = 0.22 \text{ in of } H_2 O$$

Adding other pressure losses might increase the pressure drop to 0.35 or 0.4 in of water.

Fan Requirement:

The fan to be used in the air cooled heat exchanger should cover 40% of the area to be cooled. Thus

$$A_{fan} = 0.4 \times 4 \text{ ft} \times 3.1 \text{ ft} = 4.69 \text{ ft}^2$$

The diameter of the fan is,

$$D_{fan} = \sqrt{\frac{4 \times 4.96}{\pi}} = 2.51 \text{ ft} = 30 \text{ inch}$$

For a 30 inch diameter fan the power requirements using an industry standard equation is,

$$HP_{ideal} = \frac{(\Delta p_t)(CFM)(5.2)}{33,000} HP$$

Where,  $\Delta p_t = 0.4$  in of H<sub>2</sub>O

The air flow is calculated as,

$$CFM = 87.3 \frac{ft}{s} \times \frac{60s}{min} \times 3.1 ft \times 4 ft = 6500 CFM$$

The ideal fan horsepower is calculated to be 0.41 HP. At a 75% efficiency the required fan horsepower is 0.55 HP.

Summary:

The resulting design consists of a tube bundle with 3 rows (16-17-16 arrangement). A

30" diameter fan with a  $\frac{1}{2}$  or  $\frac{3}{4}$  HP motor provide the required air flow. The overall

air-side pressure drop is 0.5" of water.

# VITA

## ANURADHA MUKHERJEE

# Candidate for the Degree of

## Master of Science

## Thesis: ON FARM ETHANOL DEWATERING

Major Field: Chemical Engineering

**Biographical**:

Personal Data: Born in Jamshedpur, India to Tapan Kumar Mukherjee (father) and Shikha Mukherjee (mother) on 30<sup>th</sup> December 1982.

Education:

Received B.E, Chemical Engineering from University of Pune in 2004, first class with distinction. Completed the requirements for the Master of Science in Chemical Engineering at Oklahoma State University, Stillwater, Oklahoma in Dec 2009.

Experience:

Research Assistant, Aug 2007 – Present, Department of Chemical Engineering, Biofuels:
Detailed design of an on-farm ethanol dewatering process.
Teaching Assistant, Aug 2007-Dec 2007, Unit Operations Laboratory, Dept of Chemical Engineering, OSU, Supervision of senior students: Overall safety compliance.
Design Engineer, Feb 2007- July 2007, Mistral Air-Conditioning Services Pvt. Ltd New Delhi, India, Design of central HVAC systems.
Research Associate, Aug 2004 – Feb 2007, Home and Personal Care Research, Unilever Research India, Bangalore, Multiple projects: Water, recycling, Soil sink and Bio-Cleaning; Lab and pilot scale experimental design and execution; Experience in mathematical process modeling; Data collection, analysis and presentation

Professional Memberships: American Institute of Chemical Engineers (AIChE), Omega Chi Epsilon Honor Society
Name: Anuradha Mukherjee

Institution: Oklahoma State University

Date of Degree: December 2009

Location: Stillwater, Oklahoma

## Title of Study: ON-FARM ETHANOL DEWATERING

Pages in Study: 134

Candidate for the Degree of Master of Science

## Major Field: Chemical Engineering

## Scope and Method of Study:

Sweet sorghum offers a range of advantages due to its low water input, high carbohydrate yield and drought tolerant characteristics. The team at the School of Bio-systems & Agricultural Engineering at Oklahoma State University has demonstrated the ability to harvest, express, and ferment sweet sorghum on-farm. The fermentation product is an aqueous 6 - 10 wt % ethanol product. The high concentration of water (90+ wt %) in the fermentation product provides a strong incentive to perform on-farm dewatering to replace high costs for transportation to a central processing facility. Furthermore, to ensure successful recovery of the ethanol obtained from fermenting sweet sorghum on farm, a small scale dewatering facility needs to be established. Rigorous, first-principles simulations have been created to provide the material and energy balances required for equipment design. Based on the findings from the simulations, the detailed design of a two-column dewatering system capable of producing a 190 proof ethanol product was completed.

## Findings and Conclusions:

The design addresses contemporary issues of water requirements and waste disposal. The proposed design eliminates the need for well water make-up, required for cooling or steam generation, with air-cooling and reboiler substitutes. The system is designed to process approximately 250 gph of fermentation product in two 12-inch diameter columns, the smallest practical size that can be implemented on a farm. With a state-of-the-art demonstration unit future studies can be geared towards data generation and analysis required for economic and technical optimization of an on-farm ethanol production facility. Results from this work could have significant economic impact on agriculture. The role of the farmer will expand from feedstock supplier to that of fuel producer and increase the economic opportunities for local communities.

ADVISER'S APPROVAL: Dr. James R. Whiteley